

A sepia-toned photograph of a hand holding a small, cylindrical metal component. The component has a knurled top section and a smooth bottom section with three small holes. In the background, a large industrial machine, possibly a lathe or mill, is visible with a bright light reflecting off its surface.

*Summary of Activities*

1997

NASA's Microgravity Technology Report

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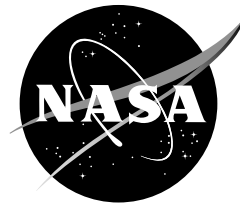
# NASA's Microgravity Technology Report

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# 1997

Summary of Activities

NASA/TM-1998-208532



National Aeronautics and  
Space Administration

George C. Marshall Space Flight Center  
Marshall Space Flight Center, Alabama 35812

# Acknowledgments

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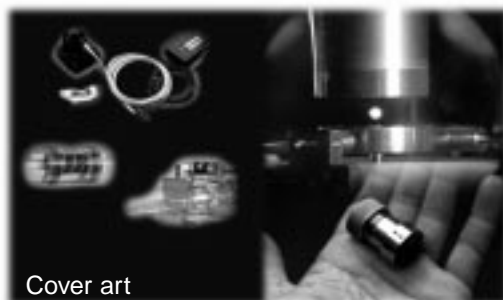
This document has been prepared by Isabella Kierk, the Microgravity Technology Development and Transfer Program manager, who would like to thank NASA field center microgravity technologists for their support and coordination of data collection. Special thanks to Martin Barmatz (the Jet Propulsion Laboratory), Dennis Morrison (Johnson Space Center), Thomas Glasgow (Lewis Research Center), and Ann Trausch (Marshall Space Flight Center) for their valuable contributions to this document.

## Descriptions of Cover Photographs

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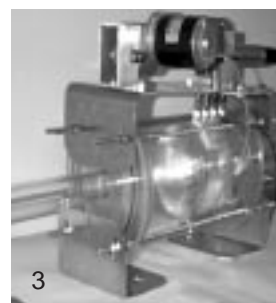
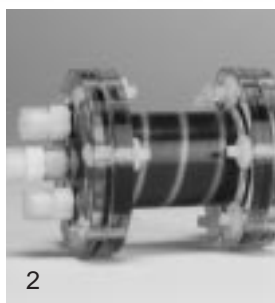
### 1. System Components for Advanced Laser Doppler Velocimetry —

These nonintrusive instruments are used to make measurements and perform analyses of data from combustion experiments.



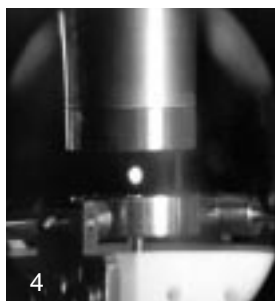
### 2. Bioproduct Recovery System cartridge —

This device allows the selective removal and preservation of molecules from space bioreactors.



### 3. Passive Free-Vortex Separator —

This device enables the separation of two-phase fluid mixtures in microgravity.



### 4. Electrostatic Levitator —

The electrostatic levitator uses static electricity inside a vacuum chamber to create a near-weightless environment for the containerless study of material properties. (Photo credit: Loral)

### 5. Magnetostrictive Low-Temperature Actuator —

This device is capable of generating mechanical movement and producing force at cryogenic temperatures.

# Table of Contents

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1.0	INTRODUCTION .....	1
	1.1 Purpose .....	1
	1.2 Scope .....	1
	1.3 Background .....	1
2.0	MICROGRAVITY RESEARCH DIVISION TECHNOLOGY ASSESSMENT AND SELECTION POLICY .....	2
	2.1 Identification of Technology Requirements and Priorities .....	2
	2.2 Evaluation of Proposed Technology Developments .....	2
	2.3 Evaluation Criteria .....	2
	2.4 Metrics .....	2
	2.5 Technology Planning and Development — Coordination With Other NASA Offices, Federal Agencies, and Universities.....	3
	2.6 International Cooperation .....	4
	2.7 ATD Program .....	4
	2.8 Technology Transfer Programs .....	5
	2.9 Technology Infusion for ISS Research Facilities .....	10
3.0	MICROGRAVITY SCIENCE RESEARCH PROGRAMS AND RELATED TECHNOLOGY DEVELOPMENTS AND REQUIREMENTS.....	11
	3.1 Biotechnology.....	11
	3.2 Combustion Science .....	12
	3.3 Fluid Physics .....	13
	3.4 Fundamental Physics .....	14
	3.5 Materials Science .....	15
	3.6 Multidiscipline Technologies .....	16
APPENDIX A.	TECHNOLOGY DEVELOPMENTS AND REQUIREMENTS DESCRIPTIONS .....	17
	A.1 Biotechnology.....	17
	A.2 Combustion Science .....	21
	A.3 Fluid Physics .....	23
	A.4 Fundamental Physics .....	24
	A.5 Materials Science .....	30
	A.6 Multidiscipline Technologies .....	35
APPENDIX B.	FY 1997 ADVANCED TECHNOLOGY DEVELOPMENT PROGRAM AND PROJECT DESCRIPTIONS.....	37
	B.1 Program Description .....	37
	B.2 Project Descriptions .....	37
APPENDIX C.	ABBREVIATIONS AND ACRONYMS .....	72

# 1.0 Introduction

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## 1.1 PURPOSE

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The purpose of this document is to update the Microgravity Research Program's (MRP's) technology development policy and to present and assess current technology-related activities and requirements identified within its research and technology disciplines. This document serves as a prime source of information for the National Research Council, the Administration's Office of Science and Technology Policy, the Office of Management and Budget, and Congress to promote technology as a means for scientific and economic growth.

## 1.2 SCOPE

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This document covers technology development and technology transfer activities within the MRP during fiscal year 1997. This report also describes the recent major tasks under the Advanced Technology Development Program and identifies current technology requirements. This document is consistent with the Human Exploration and Development of Space Strategic Plan and is updated annually to reflect changes in the MRP's new technology activities and requirements.

## 1.3 BACKGROUND

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Microgravity, the state of low gravity found in orbiting spacecraft, has unique characteristics that allow investigations of phenomena and processes that are difficult or impossible to study on Earth. NASA's Microgravity Research Division sponsors research on important biological, chemical, and physical processes in a microgravity environment in five major areas: biotechnology, combustion science, fluid physics, fundamental physics, and materials science. Technology plays a critical role in all microgravity research areas.

## 2.0

# Microgravity Research Division Technology Assessment and Selection Policy

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### 2.1 IDENTIFICATION OF TECHNOLOGY REQUIREMENTS AND PRIORITIES

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In fiscal year (FY) 1997, the third annual technology survey was conducted at the NASA field centers, providing data for ongoing and proposed technology activities and identifying technology requirements through FY 1996. The results of this survey have been documented in *NASA's Microgravity Technology Report — Summary of Activities in FY 1996*.

The Advanced Technology Development (ATD) Program presented technology poster sessions during all of the microgravity discipline science workshops and technology conferences during FY 1997, providing microgravity scientists and technologists with an opportunity to communicate the results of their work and to identify new technology needs. There are plans to stage an annual microgravity technology workshop to further improve these communications and to consider other effective mechanisms to identify and prioritize new technology requirements. These requirements will help the researchers working under the ATD Program and the discipline science programs to focus their efforts on the Human Exploration and Development of Space (HEDS) enterprise and the Microgravity Research Program (MRP) priorities. They will also help the program evaluators to select the best technology proposals for funding. The first of these workshops is planned for September 1998.

### 2.2 EVALUATION OF PROPOSED TECHNOLOGY DEVELOPMENTS

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In FY 1997, the Microgravity Research Division (MRD) participated in the evaluation of proposed technologies under the ATD Program.

A total of 34 two-page concept papers were submitted. Fourteen proposers were invited to submit full proposals; 12 of these proposals were selected for technology development in FY 1998. The final selections are listed below:

- Space Bioreactor Product Recovery and Media Reclamation Systems
- Development of an Electrostriction Cold Valve-Phase Separator

- Small High-Resolution Thermometer
- Space-Qualifiable Magneto-Optical Trap
- Compact Video-Microscope Imaging System
- A Robust Magnetic-Resonant Imager for Ground- and Flight-Based Measurements of Fluid Phenomena
- A Pulsed Tunable Laser System for Combustion
- Vibration Isolation and Control System for Small Microgravity Payloads
- A Diffractometer for Reciprocal Space Mapping of Macromolecular Crystals
- Transient Torque Viscometer for Viscosity and Electrical Conductivity Measurements
- Solid-Liquid Interface Characterization Hardware
- Quantitative Computed Tomography for Materials Science

### 2.3 EVALUATION CRITERIA

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Criteria used to evaluate technology developments within the MRP in FY 1997 included the following standards:

- Degree of relevance to the technology needs of the MRP and the HEDS enterprise
- Potential to enable new types of important microgravity investigations
- Potential for successful accomplishment based on proposed schedule and budget, qualifications and experience of the research team, and adequacy of facilities
- Potential for technology transfer to the private sector or other users
- Uniqueness or likelihood that the ATD project would not be accomplished without MRD support

### 2.4 METRICS

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The following metrics were used in microgravity research and technology programs in FY 1997:

- Number of technical/scientific publications
- Number of citations in technical/scientific literature
- Return on investment (funds gained from the program/technology vs. funds invested)
- Number of patents (applied for and awarded)
- Number of corporate agreements (technology transfer)



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- Number of flight programs using the evaluated technology
  - Number of principal investigators and co-investigators working under the program
  - Number of graduate students funded under the program
  - Number of graduate degrees granted under the program
  - Size of the portfolio (total dollars spent on research/technology development and transfer partnerships by NASA and its partners)

Metrics for the ATD Program and technology transfer programs are listed in sections 2.7 and 2.8 of this report.

## **2.5 TECHNOLOGY PLANNING AND DEVELOPMENT — COORDINATION WITH OTHER NASA OFFICES, FEDERAL AGENCIES, AND UNIVERSITIES**

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During FY 1997, continued collaborations of NASA and National Institutes of Health (NIH) biotechnology programs focused on the following areas:

- Establishment of joint NASA-NIH centers that will accelerate the transfer of NASA technology and allow its application to biomedical research
- Development of advanced tissue culturing technology and application of this breakthrough technology to biomedical research and developmental biology
- Development of advanced protein crystallization technologies to further structural biology and drug design to fight a number of diseases
- Development of technology for the early detection of cataracts

The NASA-NIH collaboration during FY 1997 continued to offer an opportunity to address the technical challenges of three-dimensional tissue growth, crystallization of high-quality protein crystals, and the early detection of cataracts, by supporting multidisciplinary research teams. These research teams allow the best American scientists and bioengineers to address these complex problems and accelerate development of related technologies.

Work at Lewis Research Center (LeRC) demonstrated that laser light scattering technology developed under a past ATD project could be used to measure the size distribution of a protein in the eye that is related to the early development stages of cataracts; discussions with managers from the NIH National Eye Institute (NEI) led to the decision to proceed with development of a prototype diagnostic tool for cataracts. In FY 1997, the MRD continued to work with the NEI under an interagency agreement for cooperative efforts in this area. Subsequent to successful demonstration, the NEI is interested in obtaining the technology for use in a large-scale clinical trial. Microgravity researchers are also collaborating with researchers at the NEI using protein crystal growth technology to determine the structures of proteins related to the signal pathway for sight, through a joint program between NASA, the NIH, and Eli Lilly and Company.

A year ago, NASA and the NIH signed an agreement to facilitate the development of a new X-ray technology with the potential to improve scientific research and enhance quality of life through better medical imaging instruments. The collaborative research agreement takes X-ray technology recently developed by Marshall Space Flight Center (MSFC), along with X-Ray Optical System, Inc., of Albany, New York, and the Center of X-Ray Optics, of the State University of New York, Albany, and enhances its imaging capabilities for a variety of commercial uses. Expected applications in scientific research and medicine include better manufacturing control for semiconductor circuits and better imaging for medical techniques, such as those used in mammography and forensics. Once developed, the X-ray device will enhance a researcher's ability to determine difficult protein structures at a faster pace, which is critical to new drug design. The NASA-developed X-ray technology is capable of generating beams with more than 100 times the intensity of conventional X-rays. At the heart of the NASA technology is a new type of optics for X-rays called capillary optics. The X-rays can be controlled by reflecting them through many thousands of tiny curved channels, or capillaries. This technique is similar to the way light is directed through fiber optics. The high-intensity beams will permit scientific and medical research to be performed in less time with higher accuracy, and could permit the use of smaller, lower-cost, and safer X-ray sources. NASA's contribution to the agreement is sponsored by the

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Microgravity Research Program Office at MSFC for the Office of Life and Microgravity Sciences and Applications at NASA headquarters. The technology will eventually be applied to research on the space shuttle and the International Space Station (ISS). The agreement between NASA and the NIH will be effective through September 30, 1999.

In FY 1997, researchers from the ATD Program continued to collaborate with the National Institute of Standards and Technology and with Sandia Laboratory on a room-temperature superconducting quantum interference detector project in the laser-induced incandescence technology area, and with the Tennessee Space Institute and the University of Chicago in stereo-imaging velocimetry technologies. Other university participants in the ATD Program included the University of Delaware in a free-float trajectory management project, Stanford University in a high-resolution pressure transducer and controller project, the University of Alabama in multicolor holography and real-time X-ray microscopy, and Case Western Reserve University in surface light scattering.

## **2.6 INTERNATIONAL COOPERATION**

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In FY 1997, the MRD held or participated in several international meetings and conferences, which are listed in the FY 1997 annual report.

As more cooperative technology development opportunities present themselves and more data become available supporting benefits resulting from the individual international agreements, an integrated international technology development program can be established. With the focus on enabling and enhancing microgravity science, this program will be tailored very carefully to assure maximum return on investment and yet preserve the national interest and competitiveness of all participating countries.

### **The Netherlands**

An ATD project researcher from LeRC spent the summer in residence at the Van der Waals–Zeeman Laboratory at the University of Amsterdam, working in close cooperation with a research faculty member there. The focus of their work was the use of a new version of LeRC's anti-slosh optical train and miniaturized surface light scattering instrumen-

tation for a binary fluid experiment inside a modified critical point facility. This experiment was one of several critical fluids experiments performed at the Van der Waals–Zeeman Laboratory, which is a world leader in the study of critical phenomena. The collaborative work between NASA and the Van der Waals–Zeeman Laboratory produced several synergistic developments. Primary among these is a new cross-correlation detection technique, which supersedes the anti-slosh optical train for experiments in which the fluid can be contained in a transparent, cylindrical cell. The new technique is simple to use and align, and it works with fluids that range from transparent to opaque. The researchers have filed invention disclosure documents on this idea. Researchers at the Van der Waals–Zeeman Laboratory are pursuing the possibilities of using the new cell design/cross-correlation surface light scattering technique, as well as another new multiple scattering suppression technique that was invented at LeRC. The latter technique uses cross correlation on bulk fluids with laser light scattering to suppress multiple scattering effects and thus extend the concentration (turbidity) range of laser light scattering. This technique will significantly extend the available concentration range for both colloid and critical fluid studies. Both of these ideas are being considered for future flight experiments.

Laser light scattering equipment that was developed under the ATD Program has been flown by NASA. Recent laser light scattering and surface light scattering ATD project innovations are being considered for future use by both NASA and the European Space Agency (ESA) in several upcoming and potential flight experiments in such areas as critical phenomena, colloidal studies, foams, magneto-rheological studies, glass transitions, and surface boundary conditions.

## **2.7 ATD PROGRAM**

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The primary goal of the ATD Program is to develop technology that will enable new types of scientific investigations by enhancing the capability and quality of the MRP's experimental hardware or by overcoming existing technology-based limitations identified in the program. This goal has been augmented in FY 1997 to satisfy technology needs of the HEDS enterprise. The ATD Program achieves these goals by funding selected technology development projects at NASA centers.



The ATD Program is intended to fund technology development through initial demonstration of feasibility and suitability for use in either the ground- or flight-based programs. Once a sufficient level of maturity is demonstrated, further development is the responsibility of an approved ground- or flight-based project team. Examples of technologies suitable for development under the program include, but are not limited to, the following: diagnostic instrumentation and measurement techniques that will benefit microgravity research; observational instrumentation and data recording methods, such as high-rate/high-resolution video; technology to enhance experiment operations, such as advanced data handling, control, decision aids, and communication (e.g., remote operations); and technology designed to advance the state of the art in hardware design, enabling new types of scientific investigations.

ATD projects may also be used to perform the following types of activities:

- Evaluation of the capabilities of existing technologies to solve problems inherent in a generic class of microgravity hardware
- Assessment of the current state of the art in a particular technology that promises to enhance or expand current capabilities
- Development of novel technologies or adaptation of existing hardware or methodologies to meet the future needs of the microgravity science program, or to expand the potential for experimentation
- Demonstration of the technical maturity and readiness of technology for incorporation into ground- or flight-based experiments

Current ATD Program projects and their progress in FY 1997 are described in the *ATD Program 1997 Annual Update* and also in Appendix B of this report. Descriptions of the ATD Program structure and process can be found in the "Program Plan" section of the *Annual Update*.

TABLE 1  
ATD PROGRAM METRICS

	FY 1994	FY 1995	FY 1996	FY 1997
Total funding	\$1,679,000	\$2,714,000	\$2,468,000	\$2,658,000
Number of tasks	13	15	14	17
Proceedings papers	6	9	2	1
Journal articles	6	10	7	19
NASA Technical Briefs	1	5	5	8
Technical presentations	11	11	9	9
Patents	0	1	2	1p*

\*p = pending

## 2.8 TECHNOLOGY TRANSFER PROGRAMS

Highlights of technology transfer programs in FY 1997 included the following activities:

### Bioreactor Technologies

To accelerate the pace of technology transfer begun under the NASA-NIH interagency agreement, two multidisciplinary research centers are currently supported: the Massachusetts Institute of Technology in Cambridge, Massachusetts, and the Wistar Institute in Philadelphia, Pennsylvania. Through NASA-NIH cooperation, NASA has funded approximately 28 research proposals and has also supported NIH-approved researchers in testing tissue samples in NASA bioreactors at Johnson Space Center (JSC). This has proven to be a very important undertaking in encouraging researchers to test NASA technology and in gaining acceptance in the larger biomedical community. NASA and the NIH have established a bioreactor laboratory under a cooperative effort that was initiated with the National Institute of Child Health and Human Development in the fall of 1994.

As of 1997, great strides continue to be made as a result of this collaboration. The transfer of NASA's bioreactor technology will promote AIDS research using cultures of human tonsil, lung, adenoid, and lymph node tissues to assess the infectivity of the HIV virus on these tissues.

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In addition, new technologies in microencapsulation and biomolecule recovery are currently being developed.

### **Advanced Diagnostics for Combustion**

While recent advances in optical and solid-state device technologies continue to place previously intractable concepts within reach of combustion science applications, the challenge remains to develop and demonstrate these technologies in microgravity combustion science experiments. Over the past decade, technology development efforts within the microgravity combustion science discipline have focused primarily on nonperturbative optical techniques for several reasons. The absence of the relatively vigorous action of buoyant convection and the interest in a variety of near-limit conditions combine to promote nonintrusive optical diagnostics as the preferred approach. In addition, optical methods are capable of high spatial and temporal resolution, features that are extremely advantageous for flight applications due to limitations on both experiment duration and expendable resources. Present efforts to develop diagnostic capabilities suitable for flight applications address the specific needs of new and ongoing investigations in the microgravity combustion science program and focus especially on the areas of need that are least mature in terms of use in the MRD's flight program.

Taken as a whole, the list of microgravity combustion science investigations contains a broad representation of distinct technical areas, including droplet combustion, flame spread and flammability limits, diffusion flames (both laminar and turbulent), premixed gaseous combustion, and soot morphology and radiation. The commensurate list of individual investigators provides an equally varied range of diagnostic capabilities and experience; some enter with only rudimentary measurement strategies, while others bring state-of-the-art capabilities to their projects. The former demonstrate a continuing dependence on NASA expertise to provide the appropriate diagnostic systems. While the latter are experts in normal-gravity laboratory studies, the transition to reduced gravity represents a challenge for which NASA is uniquely equipped both in terms of resources and expertise. Thus, there exists a significant demand to satisfy the measurement requirements of these investigators in a manner that will ultimately be suitable for spaceflight utilization.

### **Laser Light Scattering**

Laser light scattering is a technique generally used to characterize very small particles by size, shape, and tendency to associate. Researchers at LeRC working on an ATD project have developed a miniature laser light scattering unit based on fiber-optic technology. This unit has been developed to study microscopic particle behavior in fluid flow studies in space — a technology important to fundamental research, medical research, and materials processing. Much of the same technology will be used in a flight experiment scheduled to fly on the Russian space station, *Mir*. The experiment will use colloidal particles to study the process of gelation. Laser light scattering experiments will also be accommodated in the Fluids and Combustion Facility for the ISS, which LeRC is helping to design.

A number of successful respondents to the fluid physics NASA Research Announcement have indicated in their proposals the intention of using light scattering instruments. The laser light scattering technology has also proven its value in protein crystal growth processes. The LeRC Laser Light Scattering Laboratory is designing a multiangle laser light scattering system using a Mach Zender interferometer for studying macromolecular growth phenomena of protein crystals in gel media. This is an ongoing project with the Center for Macromolecular Crystallography at the University of Alabama, Birmingham. Also, several state-of-the-art, fiber optics-based, static light scattering systems have been developed for the ongoing protein crystal growth research at Mississippi State University.

Outside of NASA, the most interest in this compact, low-power, solid-state light scattering instrument has been directed toward its potential use as a diagnostic for cataracts (see section 2.5). The instrument appears to be ideal for quantification of cataract development and may lead to a cure for this common disease. This aspect of laser light scattering is being very ably pursued in cooperation with the NIH. The MRP is currently working with the NIH National Eye Institute to transfer NASA technology involving the use of laser light scattering to detect early signs of the onset of cataract formation. In another NIH-funded project, International Development and Energy Associates of Beltsville, Maryland, a commercial concern, has asked LeRC to design an instrument for ground-based study of protein crystal growth.

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Early in the laser light scattering ATD work, LeRC arranged with Brookhaven Instruments Company the manufacture of a compact correlator. The correlator, which was at that time a steamer trunk-sized computer, was reduced to a single, personal computer-insertable board.

### **Surface Light Scattering**

In this process, thermally activated ripples on the surfaces of liquids are used to measure viscosity and surface energy. This technology is being advanced by a team of collaborators from LeRC and Case Western Reserve University. The researchers can now reliably measure surface energy and viscosity rapidly and noninvasively.

In FY 1997, researchers demonstrated that high-amplitude capillary waves and mechanical or capacitively induced vibrating liquid-surface phenomena can be studied using a Mach Zender interferometer-type vibrometer. The rich power spectrum of vibrational modes attained from the absolute measurement of the system could also correlate to the viscosity of the liquid. In addition, this system has been demonstrated on different types of levitated liquid drops at room temperature. A feasibility study and demonstration is under way for the measurement of viscosity of undercooled molten materials during containerless processing. LeRC's Laser Light Scattering Laboratory is assisting scientists from the Containerless Research Institute, a research and development-based company in Evanston, Illinois.

### **Dynamic Light Scattering With Suppression of Multiple Scattering**

In this project, an instrument that uses cross correlation of scattered laser light to discriminate against multiply scattered photons and select singly scattered light will be developed and demonstrated. This will allow particle size characterization in media so turbid that the conventional light scattering methods yield significantly undersized results.

Preliminary work was reported at several national and international conferences and meetings. As a result of these meetings, a domestic company expressed specific interest in licensing the multiple scattering suppression technology. Isolab, Inc., of Akron, Ohio, is a clinical diagnostic test kit manufacturer. Among several products manufactured by the company are packaged samples of gold colloidal particles labeled with monoclonal antibodies

specific to various hemoglobin variants. These are used by several clinical and chemical product customers to determine or confirm the presence of variants such as hemoglobin S in samples from newborn screening programs and in adult patient populations. One problem Isolab is facing with this particular product is controlling the size of the gold particles during the manufacturing process. The Laser Light Scattering Laboratory at LeRC was able to help the company overcome this problem by providing them with the technology of using laser light scattering to measure the gold particle size and size distribution. At the request of the vice president of research and development, a research staff from Isolab visited the Laser Light Scattering Laboratory to determine the feasibility of using this technology. They were able to run a number of samples as a preliminary test measurement, and data analysis and reduction were provided. Currently, available commercial particle sizing and characterization light scattering units are quite expensive for small research companies like Isolab. One of the objectives for the current ATD project Laser Light Scattering with Multiple Scattering Suppression is the development of an alternative way of creating a correlator card and a fiber optics-based light scattering unit relatively inexpensively. Availability of the system is expected before the completion of this new ATD, which will end in the year 2000.

The technologies supporting many varieties of microgravity flight- and ground-based experiments are ultimately used in different types of coherent radiation (e.g., lasers). Many experiments rely strictly on the optical characteristics of laser devices. Due to the rapid advancement of semiconductor laser technology, many gas and chemical lasers have been replaced with semiconductor lasers. The many advantages of semiconductor lasers include efficiency, power consumption, and, primarily, the miniature size and light weight of the instruments. Due to these features, many microgravity experiments that require laser devices now incorporate semiconductor lasers. On the other hand, thermal control and current control of the laser devices are crucial to their proper operation, since the optical characteristics of the semiconductor laser devices strongly depend on these two factors. Commercially available laser diode controllers meet these requirements, but they are available only with a standard-sized package unit, which weighs more than 20 pounds and generally has the dimension of 1' x 1' x 8". The Laser Light Scattering Laboratory

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has initiated the development of a PC card-type laser diode controller. This type of controller has never been developed before. Currently, LeRC's Laser Light Scattering Laboratory is collaborating with a well-known laser device company in developing this controller board, which will become available before the end of 1997. Several attractive features include a single, lightweight, compact PC card; parameters for the proper operation of the lasers easily controlled from a PC Windows-based program; and a plug-and-play type of board. Laser parameters such as power, current, and temperature will be data-logged automatically in the computer. The transfer of this technology will greatly impact the semiconductor laser market, since this controller card will fit into any type of PC and is considerably cheaper than other laser controller alternatives.

One of several methods of protein crystallization is the electrophoretic method, in which salts and precipitants are electrophoretically delivered into a macromolecular solution in a controlled, reproducible way. At the same time, the supersaturation and crystallization stages must be continuously monitored in order to dynamically control the electrophoresis process. A good method to accomplish this is to probe the solution with a static laser light scattering system. LeRC has been collaborating with Bio Space International, Inc., to implement the dynamic light scattering system into electrophoretic devices. The design is based upon the miniaturization of the fiber optics-based dynamic/static laser light scattering system that is part of the current and previous ATD laser light scattering research projects.

#### **Microwave Furnace Facility**

A team of researchers at the Jet Propulsion Laboratory (JPL) has developed an advanced technique for processing materials amenable to microwave heating. This technique has the novel effect of producing volumetric heating in many important materials. The new heating technique can be used for contained or containerless processing of materials in a space environment. Under the ATD Program, researchers have also developed advanced theoretical models that define experimental conditions required for improved processing of ceramics. These analytical models can accurately describe the microwave-material interactions taking place in the furnace and can predict the temperature profiles within spherical and cylindrical samples. The facility also allows heating of materials using very precise time-temperature profiles. The researchers

at JPL are in the process of transferring this technology to the private sector.

During 1997, JPL researchers have continued their teaming arrangements with several U.S. companies under the JPL Technology Affiliates Program. They successfully demonstrated the chemical vapor deposition of silicon carbide on four moving carbon core fibers using a new microwave method. This project, which is in collaboration with 3M, is designed to reduce the cost of producing high-strength, high-temperature fibers for fabricating low-density composites. They have also teamed up with several oil and gas industrial partners to develop a new diamond cutter for drilling at higher temperatures in hard rock. This year's project activities demonstrated strong braze joints between diamond disks and tungsten carbide using the selective heating features of microwaves. A new task was also initiated to develop a microwave facility for efficiently melting plastics at rates greater than 100 kg/hr.

#### **Electrostatic Levitation Facility**

The Electrostatic Levitation Facility was developed under the MRD's containerless materials processing program. In this facility, a molten sample is isolated from container walls in a high vacuum by electrostatic levitation. Under such a condition, melts can achieve deeply undercooled states, reaching metastable liquid or solid phases. Some important applications of this facility might be in the development of glass-forming alloys and the measurement of various thermophysical properties of refractory materials such as the titanium- or nickel-based alloys that are known to be highly reactive with most crucibles. Built around this electrostatic levitator are several novel, noncontact diagnostic techniques that allow the measurement of various thermophysical properties of molten metallic alloys and semiconductors. Exceeding capabilities of conventional containerless methods, this facility can measure accurate values of density, thermal expansion, surface tension, viscosity, specific heat capacity, and emissivity.

In FY 1997, scientists at JPL pursued technology transfer activities with three companies under Technology Cooperation Agreements (TCAs). The TCA on Thermophysical Property Measurements of Glass-Forming Alloys, with Amorphous Technologies International, has been

completed. Thermophysical properties have been measured during glass-formation processes for glass-forming metallic alloys with several different compositions. The effect of surface oxides on glass formation has also been studied. Density, hemispherical total emissivity, and specific heat capacity have been measured for alloys over temperature ranges from room temperature to above their melting temperatures. Techniques that measure the surface tension and viscosity of low-viscosity molten alloys have been developed and are being applied to glass-forming alloys. The feasibility of developing these techniques for high-viscosity liquids has been established. Nucleation behaviors (including the time-temperature transformation curve) of a set of pure alloys and a set of commercial-grade alloys have been investigated, and critical cooling rates have been measured.

The TCA with Monsanto Electronic Materials Corporation (MEMC) on Thermophysical Properties of Molten Silicon has been completed with measurements taken of the thermophysical properties of pure silicon and of boron-doped silicon melts. Silicon samples provided by MEMC have been processed for measurements of density and specific heat to hemispherical total emissivity, and good results have been obtained. The most noteworthy result was the measurement of the density

of molten silicon over 300 K, which is close to the melting temperature of silicon. Density values of solid and molten silicon at the melting temperature are valuable input parameters for the computational modeling of crystal growth processes.

The TCA on Commercialization of the High-Temperature Electrostatic Levitator (HTESL) with Theta Industries, Inc. (TII), has been completed along with a feasibility study for commercializing the HTESL. As TII continues their effort to commercialize the HTESL, future cooperation with JPL looks promising.

### Microencapsulation of Drugs

Microgravity research has resulted in the development of a new drug delivery system consisting of multilayered microcapsules that resemble miniature liquid-filled balloons. The microcapsules are up to 10 times larger than white blood cells. The outer polymeric membrane of the microcapsules allows the drugs inside to diffuse out at rates controlled by the polymer composition and thickness. The initial microcapsules were designed for "chemoembolization" treatment of vascular tumors. In FY 1997, the flight prototype of the Micro-g Encapsulation Processing System was delivered to JSC for manifestation on the first Utilization Flight (UF-1) for the ISS.

TABLE 2  
FY 1997 TECHNOLOGY TRANSFER PROGRAM METRICS

Activity	Center	TCAs*	CRADAs*	Transfer to Flight Project	Other Contracts	Patents
Laser Light Scattering	LeRC	0	0	2	0	0
Stereo Imaging	LeRC	0	0	1	1	0
Surface Light Scattering	LeRC	0	0	0	0	1p**
Electrostatic Levitation	JPL	3	0	0	2p	0
Microwave Processing	JPL	0	0	0	1	1, 1p
Microencapsulation of Drugs	JSC	0	0	1	0	1, 3p
Totals		3	0	4	2, 2p	2, 5p

\* TCA = Technology Cooperative Agreement

\* CRADA = Cooperative Research and Development Agreement

\*\* p = pending



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## 2.9 TECHNOLOGY INFUSION FOR ISS RESEARCH FACILITIES

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The MRP conducted workshops in January 1997 to explore technology challenges for microgravity research facilities and to invite non-NASA organizations to contribute solutions. This effort was co-sponsored by the associate administrator of the Office of Life and Microgravity Sciences and Applications (OLMSA), Arnauld Nicogossian, and the associate administrator of the Office of Space Flight, Wilbur Trafton. Over 150 workshop attendees from NASA and other organizations identified those technology items with solutions that could be pursued jointly, using Space Act Agreements and other similar agreement instruments. The Gravitational Biology and Human Health and Performance divisions of OLMSA held their own workshops.

After completion of the workshops, the divisions of OLMSA were directed to work cooperatively with the Gravitational Biology and Human Health and Performance divisions, and also with the appropriate NASA centers, to coordinate their findings and to identify those areas in which mutual cooperation was valuable. This coordination was completed, and the findings were presented to both associate administrators and endorsed for incorporation. It was recommended that the Technology Infusion content be incorporated into the ISS Program Office's Pre-Planned Product Improvement Program. This incorporation is to be achieved in FY 1998. This content will also be provided to NASA's FY 1998 Cross-Cutting Technologies Program, administered by the Office of Space Flight.

The Technology Infusion content is comprised of the following sections: Cross-Discipline Technology Areas in common with each OLMSA division; Life Sciences, which is inclusive of technologies of value to the Gravitational Biology and Human Health and Performance divisions; and a section inclusive of the Microgravity Research Program and the Space Product Development Program.

The point of contact for more information regarding the Technology Infusion Program is Ann Trausch at MSFC.



## 3.0

# Microgravity Science Research Programs and Related Technology Developments and Requirements

This section lists major technology developments in fiscal year 1997 accomplished through research programs and under the Advanced Technology Development (ATD) Program, as well as the current new technology requirements dictated by microgravity science needs. Brief descriptions of the developed technologies and new requirements are included in Appendix A.

## 3.1 BIOTECHNOLOGY

The goal of the microgravity program in biotechnology is to use the space environment to (1) identify and quantitatively understand protein structure and the processes controlling protein crystal growth; (2) assemble human cells into functional tissues for research and treatment of disease; and (3) separate components of complex mixtures. Under this program, NASA sponsors research leading to improvements in the control and yield of techniques in these areas and contributes to major development of medical, pharmaceutical, and agricultural technologies and products. Marshall

Space Flight Center is the lead center in the areas of protein crystal growth and separation technologies and is supported by Johnson Space Center in the areas of bioreactor and tissue culture technologies.

### 3.1.1 Technologies

The major technologies developed in biotechnology are listed in Table 3. For details, see Appendix A, section A.1.1.

### 3.1.2 New Technology Requirements

Priority new technology requirements are listed below. See section A.1.2 in Appendix A for details.

- 3.1.2.1 A new optical technique for protein crystal growth diagnostics
- 3.1.2.2 Development of improved rotating-wall, perfused-vessel bioreactors
- 3.1.2.3 Development of alternate bioreactor systems for culturing mammalian cells and tissues in space

TABLE 3  
TECHNOLOGIES OF THE MICROGRAVITY BIOTECHNOLOGY PROGRAM

	Technology	Start Date	End Date
3.1.1.1	Protein Crystal Growth Vapor-Diffusion Flight Hardware and Facility	1992	1997
3.1.1.2	Protein Crystal Growth in Microgravity	1992	1998
3.1.1.3	Membrane Transport Phenomena	1995	1998
3.1.1.4	Enhanced Dewar Program	1995	1999
3.1.1.5	Investigation of Protein Crystal Growth Mechanisms in Microgravity	1992	1998
3.1.1.6	Advanced High-Brilliance X-Ray Source	1993	1997
3.1.1.7	Rotating-Wall, Perfused-Vessel Bioreactor Technologies	1987	1997
3.1.1.8	Experimental Control Computer System	1992	1998
3.1.1.9*	Crystal Growth Instrumentation Development: A Protein Crystal Growth Studies Cell	1994	1997
3.1.1.10	Microencapsulation of Drugs	1996	2000
3.1.1.11*	Space Bioreactor Bioproduct Recovery System	1996	2000
3.1.1.12	Sensor Technologies	1994	2000
3.1.1.13*	A New Ultrahigh-Resolution Near-Field Microscope for Observation of Protein Crystal Growth	1997	2000

\* Developed under the Advanced Technology Development (ATD) Program

- 3.1.2.4 Cell and tissue metabolism sensor and control subsystems
- 3.1.2.5 Advanced media/nutrient supply and replenishment subsystems
- 3.1.2.6 Development of microgravity-based bioreactor systems technologies
- 3.1.2.7 Cell/tissue oxygenation and waste gas removal subsystems
- 3.1.2.8 Development of a thermally controlled sample/experiment storage system
- 3.1.2.9 Bioreactor technologies: culture systems for cellular radiation studies
- 3.1.2.10 Microencapsulation of drugs: fluid manipulations

## 3.2 COMBUSTION SCIENCE

The objectives of the microgravity program in combustion science focus on obtaining deeper understanding of combustion processes, including ignition, propagation, and extinction of various types of flames under low-gravity conditions. This program brings fresh insights to important problems, such as fire safety in space and on the ground. Since combustion accounts for a preponderance of the world's electrical power generation and provides the energy for nearly all modes of transport for goods and people, including spacecraft, this program also has significant potential for increasing energy utilization efficiency and reducing the production of combustion-generated pollution. In addition, combustion processes are now being used in the synthesis of novel materials. Microgravity research in this area offers potential for understanding such processes and for replacement of current trial-and-error approaches. Lewis Research Center is the lead center for combustion science research.

### 3.2.1 Technologies

The major technologies developed in combustion science are listed in Table 4. For details, see Appendix A, section A.2.1.

### 3.2.2 New Technology Requirements

Priority new technology requirements are listed below. See section A.2.2 in Appendix A for details.

- 3.2.2.1 Soot temperature measurements using pyrometric techniques
- 3.2.2.2 Liquid surface temperature and vapor-phase concentration measurements via exciplex fluorescence
- 3.2.2.3 Liquid-phase thermometry and fluorescence of aromatics
- 3.2.2.4 Tunable pulsed laser sources
- 3.2.2.5 Compact, efficient optical frequency shifting
- 3.2.2.6 Axial velocity measurements
- 3.2.2.7 Analysis tools for multidimensional emission/transmission measurements

TABLE 4  
TECHNOLOGIES OF THE MICROGRAVITY COMBUSTION SCIENCE PROGRAM

	Technology	Start Date	End Date
3.2.1.1	Planar Illumination for Flow Visualization and Quantitative Velocimetry	1993	1997
3.2.1.2	Laser Doppler Velocimetry	1994	Ongoing
3.2.1.3*	Advanced Diagnostics for Combustion	1997	2001
3.2.1.4*	Determination of Soot Volume Fraction via Laser-Induced Incandescence	1994	1997

\* Developed under the ATD Program

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### 3.3 FLUID PHYSICS

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The objective of the microgravity fluid physics program is to conduct comprehensive research on fluid dynamics and transport phenomena in which fundamental behavior is limited or affected by the presence of gravity, and where low-gravity experiments allow insight into that behavior. For example, a low-gravity environment results in greatly reduced density-driven convection flows and allows the study of other forms of convection, such as flows driven by surface tension gradients, magneto/electrodynamics, or other interfacial phenomena. Investigations of these phenomena result in the basic scientific and practical knowledge needed to design effective and reliable space-based systems and facilities that depend on fluid processes. Another objective of the fluid physics program is to assist other microgravity disciplines, such as materials science or combustion science, by developing an understanding of the gravity-dependent fluid phenomena that underlie their experimental observations. Lewis Research Center is the lead center for fluid physics research.

#### 3.3.1 Technologies

The major technologies developed in fluid physics are listed in Table 5. For details, see Appendix A, section A.3.1.

#### 3.3.2 New Technology Requirements

Priority new technology requirements are listed below. See section A.3.2 in Appendix A for details.

- 3.3.2.1 High-resolution, high frame-rate video
- 3.3.2.2 Improved data storage and downlink technologies
- 3.3.2.3 Three-dimensional particle tracking
- 3.3.2.4 Nonintrusive digital pattern recognition and phase boundary locator

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TABLE 5  
TECHNOLOGIES OF THE MICROGRAVITY FLUID PHYSICS PROGRAM

	Technology	Start Date	End Date
3.3.1.1	Mechanics of Granular Materials	1978	1998
3.3.1.2*	Stereo-Imaging Velocimeter	1992	1997
3.3.1.3*	Laser-Feedback Interferometer	1994	1997
3.3.1.4*	Passive Free-Vortex Separator	1996	1999
3.3.1.5*	Surface Fluctuation Spectrometers	1996	1997
3.3.1.6*	Laser Light Scattering With Multiple Scattering Suppression	1997	2000

\* Developed under the ATD Program

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### 3.4 FUNDAMENTAL PHYSICS

The objective of the microgravity fundamental physics program is to provide opportunities to test fundamental scientific theories to a level of accuracy not possible in the gravity environment on Earth. This program encompasses research on transient and equilibrium critical phenomena, as well as other thermophysical measurements of interest in condensed matter physics, relativistic physics, and atomic physics using laser-cooling technologies and quantum crystals. The Jet Propulsion Laboratory is the lead center for fundamental physics research.

#### 3.4.1 Technologies

Technologies developed in the fundamental physics program are listed in Table 6. For details, see Appendix A, section A.4.1.

#### 3.4.2 New Technology Requirements

Priority new technology requirements are listed below. See section A.4.2 in Appendix A for details.

- 3.4.2.1 Digital Signal Processing (DSP) SQUID technology
- 3.4.2.2 Improved flight-quality radio frequency SQUID system
- 3.4.2.3 Improved high-resolution pressure transducer

- 3.4.2.4 Vibration isolation for cryogenic systems
- 3.4.2.5 Precise thin-film deposition on cylindrical surfaces
- 3.4.2.6 Long-lifetime cryogenics systems for the International Space Station
- 3.4.2.7 Miniaturized HRTs
- 3.4.2.8 Flight-qualified technology and instrumentation for laser cooling of atoms
- 3.4.2.9 Visual-access cryogenic system or cryogenic high-speed video camera
- 3.4.2.10 Flight-quality direct current SQUID system
- 3.4.2.11 Flight-quality DSP SQUIDS
- 3.4.2.12 Ultrahigh-vacuum production and measurement techniques
- 3.4.2.13 Multiple experiment platform for low-temperature physics experiments
- 3.4.2.14 Low-noise cryocoolers
- 3.4.2.15 Flight-quality dilution refrigerator
- 3.4.2.16 Flight-quality GdCl<sub>3</sub> thermometer
- 3.4.2.17 Flight-quality laser-cooled atom trap facility
- 3.4.2.18 Flight-qualified <sup>3</sup>He refrigerator
- 3.4.2.19 Accelerometers for use at helium temperatures
- 3.4.2.20 Fast-response high-resolution thermometry
- 3.4.2.21 Miniature high-reliability low-temperature valve
- 3.4.2.22 Flight-quality ultrastable frequency standard
- 3.4.2.23 Low-noise cryogenic instrumentation amplifiers
- 3.4.2.24 Telescience flight software tools

TABLE 6  
TECHNOLOGIES OF THE FUNDAMENTAL PHYSICS PROGRAM

	Technology	Start Date	End Date
3.4.1.1	Copper Ammonium Bromide High-Resolution Thermometers (HRTs)	1985	Ongoing
3.4.1.2**	High-Resolution Capacitive Pressure Transducer	1994	Ongoing
3.4.1.3**	Single-Electron Transistor	1995	Ongoing
3.4.1.4**	High-Resolution Thermometry and Improved Superconducting Quantum Interference Device (SQUID) Readout	1994	Ongoing
3.4.1.5	Prototype Miniature HRT	1995	Ongoing
3.4.1.6	Magnetic Levitator for Liquid Helium	1994	Ongoing
3.4.1.7*	Magnetostrictive Low-Temperature Actuators	1996	1999
3.4.1.8	Measurement of Ultralow Pressure at Low Temperature	1996	Ongoing
3.4.1.9*	Applications of Superconducting Cavities to Microgravity Research	1997	2000
3.4.1.10*	Development of an Electrostrictive Helium Valve	1997	1998
3.4.1.11	Test Facility for Space Station-Era Cryoprobes	1997	Ongoing

\* Developed under the ATD Program

\*\* Originally developed under the ATD Program and continued under the fundamental physics program

### 3.5 MATERIALS SCIENCE

The goal of the microgravity materials science program is to use microgravity to seek and quantitatively understand cause-and-effect relationships among the processing, properties, and structures of materials. Of particular interest is understanding the role of gravity-driven convection in the processing of metals, alloys, composites, glasses, ceramics, polymers, and electronic and photonic materials. This research will help scientists better understand materials and materials processing, and it may result in improvements to production methods and materials on Earth. Marshall Space Flight Center is the lead center for materials science research.

#### 3.5.1 Technologies

Technologies developed in materials science are listed in Table 7. For details, see Appendix A, section A.5.1.

#### 3.5.2 New Technology Requirement

The priority new technology requirement is listed below. See section A.5.2 in Appendix A for details.

- 3.5.2.1 Development of an optical characterization facility for D-X defects in bismuth silicon oxide

TABLE 7  
TECHNOLOGIES OF THE MATERIALS SCIENCE PROGRAM

	Technology	Start Date	End Date
3.5.1.1	Coupled Growth in Hypermonotectics	1993	1998
3.5.1.2	Orbital Processing of High-Quality Cadmium Telluride	1990	1998
3.5.1.3	Growth of Solid-Solution Single Crystals	1994	1999
3.5.1.4	Crystal Growth of II-IV Semiconducting Alloys by Directional Solidification	1992	1998
3.5.1.5	The Study of Dopant Segregation Behavior During the Growth of GaAs in Microgravity	1992	1998
3.5.1.6	Temperature Dependence of Diffusivities in Liquid Metals	1993	1998
3.5.1.7	Particle Engulfment and Pushing by Solidifying Interfaces	1993	1998
3.5.1.8	Crystal Growth of ZnSe and Related Ternary Compound Semiconductors by Physical Vapor Transport	1993	1997
3.5.1.9	Test of Magnetic Damping of Convective Flows in Microgravity	1992	1997
3.5.1.10*	Real-Time X-Ray Microscopy for Solidification Processing	1994	1997
3.5.1.11	Physical Properties and Processing of Undercooled Metallic Glass-Forming Liquids	1995	1999
3.5.1.12	Investigation of the Relationship Between Undercooling and Solidification Velocity	1996	2000
3.5.1.13	Space- and Ground-Based Crystal Growth Using a Magnetically Coupled Baffle	1996	2000
3.5.1.14	Compound Semiconductor Crystal Growth in a Microgravity Environment	1994	1999
3.5.1.15	Orbital Processing of Eutectic Rod-Like Arrays	1996	1999
3.5.1.16	Interface Pattern Selection Criterion for Cellular Structures in Directional Solidification	1996	1999
3.5.1.17	Comparison of Structure and Segregation in Alloys Directionally Solidified in Terrestrial and Microgravity Environments	1996	1999
3.5.1.18*	Advanced Heat Pipe Technology for Furnace Element Design	1994	1997
3.5.1.19*	Ceramic Cartridges via Sintering and Vacuum Plasma	1993	1997

\* Developed under the ATD Program

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### 3.6 MULTIDISCIPLINE TECHNOLOGIES

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#### 3.6.1 Technologies

Technologies developed under the ATD Program to support multiple science disciplines are shown in Table 8. For details, see Appendix A, section A.6.1.

#### 3.6.2 New Technology Requirement

The priority new technology requirement is listed below. See section A.6.2 in Appendix A for details.

##### 3.6.2.1 Miniature microscope

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TABLE 8  
TECHNOLOGIES OF MULTIPLE SCIENCE DISCIPLINES

	Technology	Start Date	End Date
3.6.1.1*	Free-Float Trajectory Management	1995	1997
3.6.1.2*	Vibration Isolation and Control System for Small Microgravity Payloads	1997	1999

\* Developed under the ATD Program

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# Appendix A

## Technology Developments and Requirements Descriptions

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### A.1 BIOTECHNOLOGY

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#### A.1.1 Technologies

Following are brief descriptions of the technologies listed in section 3.1.1. Items with an asterisk were developed under the Advanced Technology Development (ATD) Program.

##### 3.1.1.1 Protein Crystal Growth Vapor-Diffusion Flight Hardware and Facility

The objectives of this research are to provide an easy-to-use interface between ground- and flight-based protein crystal growth hardware, increase (common) availability of flight hardware, eliminate the late-access requirement, and enable individual loading by the principal investigator. The Protein Crystallization Apparatus for Microgravity was successfully demonstrated as a flight article aboard the 63rd Space Transportation System (STS-63) flight, STS-67, and STS-73 (the second United States Microgravity Laboratory [USML-2] mission). The Diffusion-Controlled Crystallization Apparatus for Microgravity was developed, constructed, and flown aboard STS-73 as part of USML-2.

##### 3.1.1.2 Protein Crystal Growth in Microgravity

Automated dynamic control of protein crystal growth using controlled vapor diffusion has been used to study the effect of varying the evaporation rate on the crystals obtained for a given solution condition. The system incorporates a static laser light scattering subsystem consisting of a laser, a photodetector, and fiber optics. The subsystem allows the evaporation profile to be modified in response to nucleation events. This system has shown that detection of nucleation and modification of the evaporation profile while the experiment is in progress can improve crystal growth results.

##### 3.1.1.3 Membrane Transport Phenomena

This investigation addresses how gravity influences fluid boundary layers that are associated with membranes and how the related membrane-mediated mass transport processes are affected. The primary variable in the proposed experiments is the convective environment within the test solutions, of which there are three modes: induced convection (gravity greater than 1 g), inhibited convection (gravity less than 1 g), and forced convection (externally applied stirring). All of the experiments will be performed for each of these three convective modes. Kinetic differences between gravimetric orientations will indicate experimental conditions for which microgravity effects are likely.

##### 3.1.1.4 Enhanced Dewar Program

This program uses batch and liquid-liquid crystallization methods to screen protein crystallization conditions. A dewar flask with small crystallization samples will be frozen on Earth and allowed to passively thaw in microgravity. The crystals will be examined by standard light, electron, and atomic force microscopy. Crystal structures will be determined and analyzed. Design and development of temperature control and monitoring enhancements to the dewar apparatus were initiated.

##### 3.1.1.5 Investigation of Protein Crystal Growth Mechanisms in Microgravity

This apparatus allows for interactive crystallization experiments on orbit via telerobotics. The Canadian-American crystallization hardware provides either liquid-liquid or mixed-batch protocols for crystallization, with a unique mixing mechanism. A laboratory-based control system is used to remotely monitor the progress of crystallization experiments and provide users on the Internet with the ability to control the monitoring camera and capture an image of the individual sample wells. The first version of the control software can be run on the World Wide Web. Dynamic light scattering experiments have begun to examine the states of aggregation of protein in bulk solution and around various faces of a protein crystal.

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#### **3.1.1.6**

##### **Advanced High-Brilliance X-Ray Source**

The primary objective of this task is to produce the first X-ray generator and Kumakhov lens system optimized in design for 8.0 KeV X-rays. This task is part of the development of an extremely bright X-ray source for the evaluation and determination of the atomic structures of crystalline matter. Potential applications include medical imaging.

#### **3.1.1.7**

##### **Rotating-Wall, Perfused-Vessel Bioreactor Technologies**

This project develops and applies slow rotating-wall, perfused-vessel bioreactor technologies to cell and tissue culture in a simulated microgravity environment on Earth and supports tissue growth in microgravity missions. These technologies include subsystems for oxygenation; media replenishment; CO<sub>2</sub> and waste product removal; temperature control; glucose, pH, O<sub>2</sub>, and CO<sub>2</sub> monitoring and control; cell/tissue sampling and preservation; and computer systems.

#### **3.1.1.8**

##### **Experimental Control Computer System**

Experimental Control Computer System is a flight-configured compact 486 CPU-based control and data acquisition system for microgravity science applications. It incorporates removable PCMCIA memory and program cards, and provides data interfaces with individual experiment hardware systems, racks, and space vehicle data buses when telemetry capability is required.

#### **3.1.1.9\***

##### **Crystal Growth Instrumentation Development: A Protein Crystal Growth Studies Cell**

A Protein Crystal Growth Analysis Chamber and associated techniques continue to be developed to study crystal growth processes and long-term storage techniques for proteins. Aspects of growth to be facilitated for study include solution concentration gradients and interfacial features of crystals under growing and etching conditions. The apparatus will aid remote manipulation of growth parameters, such as temperature, and will allow data collection. Long-term storage capability is anticipated to be of increasing importance as opportunities for longer-duration microgravity and normal-gravity experiments become increasingly available.

#### **3.1.1.10**

##### **Microencapsulation of Drugs**

Microgravity research resulted in the development of a new drug delivery system consisting of multilayered microcapsules that resemble miniature liquid-filled balloons up to 10 times larger than white blood cells. The outer polymeric membrane of the microcapsules allows the drugs inside to diffuse out at rates controlled by the polymer composition and thickness. The initial microcapsules were designed for "chemoembolization" treatment of vascular tumors. The anti-tumor drugs are delivered directly into the tumor by injecting the microcapsules into the main artery, wherein they form emboli that reduce the blood supply to the tumor and provide sustained release of cytotoxic drugs to tumor cells. A dense radio-contrast oil has been coencapsulated that makes it possible to radiographically monitor the accumulation of these microcapsules to verify that they have lodged in the tumor arterioles. So far, six different drugs have been encapsulated in microgravity, including two anti-tumor drugs, an immune stimulant, an antibiotic, a clot-dissolving enzyme, and an anti-nauseant. Crystals of drugs have been encapsulated, which means that the interior lining of the blood vessels can be protected from the sharp crystal edges by the soft, pliable outer membrane of the microcapsules. Administration of drug crystals is a way of providing maximum sustained drug release and long-term delivery of the drug from each injection.

#### **3.1.1.11\***

##### **Space Bioreactor Bioproduct Recovery System**

The purpose of this effort is to develop a bioproduct recovery system (BRS) that allows the selective removal of molecules of interest from space bioreactors, thus enhancing the productivity of those bioreactors. The BRS is designed to target specific biomolecules or waste products, to continuously adsorb and separate biomolecules from dilute bioreactor effluents, and to stabilize and preserve targeted bioproducts. The BRS will also be miniaturized to meet volume and power constraints and designed to operate in microgravity.

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### 3.1.1.12

#### Sensor Technologies

As part of the NASA/Johnson Space Center (JSC) biotechnology program, the Sensors and Controls Research and Development Laboratory is developing new, state-of-the-art sensor and control technologies that will be an integral part of NASA/JSC perfused microgravity bioreactor systems. These technologies are considered enabling for the development of fully automated tissue engineering systems that require a minimum of operator time and consumable resources while providing a physiological environment for cell and tissue culture. Critical biologic parameters to be monitored and/or controlled are concentrations of hydrogen ions, glucose, O<sub>2</sub>, CO<sub>2</sub>, biomass, glutamine, and lactate. An amperometric glucose oxidase-based biosensor previously developed for *in-vivo* glucose monitoring has been adopted and redesigned. The sensor has been integrated with a rotating-wall microgravity bioreactor to continuously and independently monitor the bioreactor glucose level. The developed sensor has been tested extensively in various bioreactor conditions. Effects of several factors, such as media pH, dissolved O<sub>2</sub>, perfusion flow rates, and long-term exposure, on sensor performance have been determined. Glucose concentrations in cell culture media have been monitored continuously over 21 days. Monitoring of pH in perfused microgravity bioreactors is performed continuously and noninvasively by the optical pH sensor developed by NASA/JSC. Cell culture media pH is measured indirectly by the sensor via detection of the light frequency transmitted through Phenol Red, a common pH indicator in cell culture media. The sensor then converts the frequency to a pH reading. The optical pH sensor has been integrated into a NASA-developed perfused microgravity bioreactor.

### 3.1.1.13\*

#### A New Ultrahigh-Resolution Near-Field Microscope for Observation of Protein Crystal Growth

The main goal of this ATD project is to build and test a new optical method for observing protein crystals as they nucleate and grow. The new technique will be based on a tapered-fiber probe in a near-field scanning optical microscope (NSOM).

### A.1.2

#### New Technology Requirements

Following are brief descriptions of the new technology requirements listed in section 3.1.2.

### 3.1.2.1

#### A new optical technique for protein crystal growth diagnostics

An optical system for observing protein crystals as they nucleate and grow is needed. Standard microscopy is limited in resolution to about a half wavelength of light, and electron and X-ray microscopy cannot overcome the diffraction limit. An NSOM has a submicron resolution and is proposed for the observation of a growing protein crystal. Resolutions of 10-nm patterns have been shown. With the use of fluorescent labels, discrimination between single molecules and aggregates can be accomplished when they are separated by as little as 40 nm. A tapered-fiber probe for immersion in growth solutions has also been proposed as an integral part of the NSOM optical system. Optimization of the system for the unique application of protein crystal growth is needed, and if successful, observation of how monomers (single molecules) orient and form into a nucleus may be possible. In addition, optical monitoring of the surface of the growing crystal may be achieved.

### 3.1.2.2

#### Development of improved rotating-wall, perfused-vessel bioreactors

Previously developed rotating-wall perfused bioreactors have been used to simulate many of the low-gravity conditions of space for ground-based mammalian tissue culturing. However, these systems are difficult to operate and maintain. Advanced rotating-wall bioreactors have been designed for improved long-term reliability and maintainability for both ground-based research and spaceflight experiments. The new systems have an enhanced ability to remove sterile samples of cells and culture medium with minimum disruption of the tissue-like spheroids of cells growing together. Improved process control parameters and reduced levels of hydrodynamic stress on the cells are accomplished by controlling the rotating speeds of the inner core and outer wall more precisely. Key design issues include sterilizable components, improved materials compatibility, and reduced cell toxicity for longer-term studies of multiple types of tissue-like cellular constructs.

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#### 3.1.2.3

##### **Development of alternate bioreactor systems for culturing mammalian cells and tissues in space**

These systems include automated Perfused Stationary Culture Systems wherein satisfaction of nutrient requirements and removal of cellular waste products is accomplished by the slow perfusion of medium through the entire culture. Cells at the distal end of the system receive the same nutrients as cells at the perfusion inlet. These flight systems can allow for long-term culture of cells that are or are not anchorage dependent. Passive Culture Systems (PCSs) include autonomous small flight hardware for maintaining living cells and performing simple, short-term experiments under microgravity conditions. The PCSs have a limited gas storage capability for O<sub>2</sub> (maximum content is about 40%) and therefore require an external gas source. Flight designs include use of off-the-shelf plastic-ware, special sterile injection and sample ports, and different sizes of culture chambers ranging from 0.8 to 4.4 liters.

#### 3.1.2.4

##### **Cell and tissue metabolism sensor and control subsystems**

Cell metabolic sensors are crucial for proper process control of the culture microenvironment within cell culture systems. Earth-based sensor systems for O<sub>2</sub>, CO<sub>2</sub>, pH, glucose, and other metabolic parameters do not function adequately in microgravity. Therefore, modifications, miniaturization, and repackaging are necessary to provide accurate real-time data for control subsystems that minimize expendables for long-duration spaceflight culture systems. Effects of space radiation on reliability and certain computer memory systems must also be assessed, and if necessary, software must be developed that can prevent loss of critical control or experimental data. The current designs include interface with laptop computers and telemetry links for program updates or malfunction diagnosis.

#### 3.1.2.5

##### **Advanced media/nutrient supply and replenishment subsystems**

Advanced media/nutrient supply and replenishment subsystems are required to

minimize fluid and mass requirements for long-duration microgravity culture systems. Dissolved waste products, proteins, and gases all must be eliminated to prevent the build up of toxic levels within the perfusion medium. Optimum control of pH normally requires periodic addition of basic chemicals and concentrated volumes of glucose and growth factors. Metabolic nutrients also must be added frequently. Diffusion or dialysis methods can be utilized; however, injection of concentrated additives must be carefully controlled by adequate mixing and dilution prior to exposure to the cells. This requires precise chemical sensors; fluid subsystems that often need refrigeration; and process control parameters to carefully control, resupply, or replenish the culture medium perfused over the cells.

#### 3.1.2.6

##### **Development of microgravity-based bioreactor systems technologies**

Development of automated microgravity-based bioreactor systems technologies for tissue engineering led to experiments in the Biotechnology Facility (BTF) on the *Priroda* module of the Russian space station, *Mir*. The BTF can support rotating bioreactor systems and other payloads within a four-module operational configuration. A gas-supply module and a refrigeration unit accompany the cell culture payload. The Biotechnology Data Acquisition Computer PCMCIA memory cards and recovery software control and operate the BTF and the tissue culture payloads.

#### 3.1.2.7

##### **Cell/tissue oxygenation and waste gas removal subsystems**

Optimum maintenance of O<sub>2</sub> levels and waste gas removal requires special subsystems that provide precise control of dissolved gases and eliminate any bubble formation, which can denature proteins in the nutrient medium. Special gas-supply and control subsystems that maximize recycling of the atmospheric gases and minimize waste gas dumps into the cabin atmosphere are required. These include provisions for sterilizing the gases before reuse in the gas exchange subsystem within the bioreactor modules.

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### 3.1.2.8

#### **Development of a thermally controlled sample/experiment storage system**

There is a need for a thermally controlled sample/experiment storage system capable of maintaining a stable thermal environment during short-duration space station power brownouts and blackouts.

### 3.1.2.9

#### **Bioreactor technologies: culture systems for cellular radiation studies**

As part of the biotechnology and radiation protection programs, there is a need to develop small cell culture systems suitable for experiments studying radiation repair under microgravity conditions. These systems will use short, penetrating electron or proton radiation and must have a very high aspect ratio so that target cells will receive an even distribution of incident radiation. The systems must be capable of maintaining the cells with nutrients and removing toxic products in such a way that the microgravity effects on cellular radiation repair will not be masked by the environmental effects of the culture system. The overall system must be designed so that known doses of radiation can be applied to multiple cultures of cells in order that the survival and recovery mechanisms can be related to the dose response of the irradiated cells. Built-in sensors, medium recirculation, and temperature and pH control must be maintained at all times by a built-in process controller. Irradiation systems must be fail-safe and suitable for use in the crew compartment of the shuttle or space station.

### 3.1.2.10

#### **Microencapsulation of drugs: fluid manipulations**

There is a need for new ways of injecting immiscible liquids into each other under controlled shear conditions and methods to transmit high-energy acoustic waves through liquids by exposure to pulsed, microwave, ultrasonic, or laser light waves. Also, new systems that can be adapted to microscopes are needed for electromagnetic field test chambers to permit image capture of field effects on microcapsules in the 5–300 micron range.

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## **A.2 COMBUSTION SCIENCE**

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### A.2.1

#### **Technologies**

Following are brief descriptions of the technologies listed in section 3.2.1. Items with an asterisk were developed under the ATD Program.

#### 3.2.1.1

##### **Planar Illumination for Flow Visualization and Quantitative Velocimetry**

Visualization of flows usually involves seeding the medium with dyes or particles. The relative intensity of fluorescence or scattering may be used to indicate the extent of mixing. By comparing two images, velocity vectors can be ascribed to the flow field, as is done in particle image velocimetry (PIV). Both gaseous and liquid flows may be visualized and quantified with appropriate seeding using a planar laser light sheet for excitation of fluorescence or scattering from the seeded species. A variation of this technique is used to detect fluorescence from aromatic species transported back to the droplet surface from the flame in droplet combustion experiments. Such transport of rather nonvolatile species may be involved in droplet microexplosions. The specific application of this technique is for PIV. A similar configuration was used to perform quantitative planar velocimetry in the Spread Across Liquids sounding rocket experiment.

#### 3.2.1.2

##### **Laser Doppler Velocimetry**

An interference pattern can be created by using crossed laser beams. A seeded particle moving across the interference fringes creates a time-varying scattering signal. The modulation frequency of the scattered signal yields the particle velocity, and hence the gas velocity, for a given light-fringe pattern spacing. Configurations employing compact solid-state laser and detector technologies have been constructed for use in the various reduced-gravity facilities. One such system was successfully applied to turbulent, premixed gaseous combustion to support a DC-9 campaign at the Lawrence Berkeley Laboratory.



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### 3.2.1.3\*

#### **Advanced Diagnostics for Combustion**

The goal of this project is to develop a series of more sophisticated measurement techniques applicable to the general area of microgravity combustion science. These techniques are intended to improve the accuracy and spatial/temporal yield of the data acquired and to extend the range of applicability and access to the relevant parameters presently inaccessible through current methods. The project is segmented into the following two principal areas of development: species identification and quantification; and flow-field diagnostics, which addresses the characterization of complex and/or turbulent flow regimes.

### 3.2.1.4\*

#### **Determination of Soot Volume Fraction via Laser-Induced Incandescence**

Laser-induced incandescence is being studied for use as a two-dimensional imaging diagnostic tool for measuring soot volume fraction for microgravity combustion research. This method would offer more detailed information about combustion processes than present line-of-sight measurements.

## A.2.2

### **New Technology Requirements**

Following are brief descriptions of the new technology requirements listed in section 3.2.2.

### 3.2.2.1

#### **Soot temperature measurements using pyrometric techniques**

The temperature of the soot within a flame may be inferred using two-wavelength pyrometry, in which radiant energy is measured at two different wavelengths. The technique, strictly passive in nature, requires only appropriately calibrated detectors. The calculated temperature of the soot reflects that of the ambient gaseous environment.

### 3.2.2.2

#### **Liquid surface temperature and vapor-phase concentration measurements via exciplex fluorescence**

Exciplex thermometry uses an additive that forms a complex when electronically excited. Since the concentration of the complex is temperature-

dependent, and since it fluoresces at different wavelengths than the original compound, the ratio of the fluorescence intensities from the parent and complex can, with suitable calibration, yield a measure of temperature.

### 3.2.2.3

#### **Liquid-phase thermometry and fluorescence of aromatics**

Liquid-phase thermometry and fluorescence of aromatics are needed to evaluate droplet surface transport and internal flow.

### 3.2.2.4

#### **Tunable pulsed laser sources**

The need exists for tunable solid-state laser sources for application to experiments involving planar laser-induced fluorescence, Rayleigh and Raman scattering, and laser-induced incandescence. Principally, such devices must achieve power conversion efficiencies commensurate with envisioned combustion research facilities planned for the space shuttle and space station.

### 3.2.2.5

#### **Compact, efficient optical frequency shifting**

Experiments providing directional optical velocity measurements would benefit from the availability of compact, efficient optical frequency shifters. Existing technologies require relatively large volumes and significant electrical power, and result in undesirable aberrations in the emitted optical beams.

### 3.2.2.6

#### **Axial velocity measurements**

Existing provisions for three-component laser doppler velocimetry measurements are implemented in the form of stereoscopic measurements. Such configurations require wide view angles, and they poorly resolve the axial component. The ability to directly measure this component is highly desirable.

### 3.2.2.7

#### **Analysis tools for multidimensional emission/transmission measurements**

Spatial correlations between state variables in multidimensional systems can be exploited to provide quantitative determinations of species concentrations and temperature. Continued efforts



are required to provide more robust analysis methods applicable to more general chemistry and physical geometries.

## A.3 FLUID PHYSICS

### A.3.1 Technologies

Following are brief descriptions of the technologies listed in section 3.3.1. Items with an asterisk were developed under the ATD Program.

#### 3.3.1.1

##### **Mechanics of Granular Materials (MGM)**

MGM is an apparatus designed to develop a quantitative understanding of the constitutive behavior of dry and saturated sand when crushed under very low confining pressures in a micro-gravity environment.

#### 3.3.1.2\*

##### **Stereo-Imaging Velocimeter**

This project will provide a method to measure three-dimensional fluid velocities quantitatively and simultaneously by mapping and tracking multiple tracer particles. The locations of the tracer particles are determined from two camera images. One use of this technology involves multipoint particle tracking during convective flow studies.

#### 3.3.1.3\*

##### **Laser-Feedback Interferometer**

This project will develop an instrument that uses a laser as both a light source and a phased detector in order to determine phenomena that are dynamic and that vary slowly over time in micro-scopic and macroscopic fields of view. This technology could have applications in several scientific fields but especially in fluid physics and transport dynamics experiments that would benefit from accurate measurements of the change in the optical path length.

#### 3.3.1.4\*

##### **Passive Free-Vortex Separator**

Future long-term experiments will require that gas-liquid mixtures be separated into single-phase states prior to reuse or recycling. The Passive Free-Vortex Separator will be developed under the ATD program to separate two-phase fluid mixtures in microgravity.

#### 3.3.1.5\*

##### **Surface Fluctuation Spectrometers**

In this project, an instrument will be developed and demonstrated that uses a cross of scattered laser light to discriminate against multiply scattered photons and select singly scattered light. This will allow particle size characterization in turbid media. Conventional light scattering methods yield significantly undersized results. This technique extends the use of laser light scattering by several orders of magnitude in concentration. The technology permits a closer approach to critical points in critical fluid experiments and allows the examination of binary mixtures in colloid suspensions. Industry interest in this technology is strong.

#### 3.3.1.6\*

##### **Laser Light Scattering With Multiple Scattering Suppression**

Multiple scattering severely limits the concentration range over which laser light scattering can be used. This ATD project provides a simple and novel optical technique that overcomes the multiple scattering effects in turbid media. This ability to separate single scattering from multiple scattering has been tested by the Laser Light Scattering Group of NASA Lewis Research Center. The researchers are presently increasing the available size and concentration range even further by combining this technique with other developments from fiber-optic light scattering spectroscopy projects. The multiple suppression technique is now capable of measuring particle sizes ranging from a few tens of angstroms, typical for small proteins, to a few microns, typical of large colloids. It has become a fundamental diagnostic tool for experiments in colloids and critical fluids, two major areas of microgravity fluid physics research.

### A.3.2

#### **New Technology Requirements**

Following are brief descriptions of the new technology requirements listed in section 3.3.2.

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### 3.3.2.1

#### High-resolution, high frame-rate video

Commercially available technology that provides a video resolution of up to 1000 x 1000 and/or a 1000 frames-per-second acquisition rate is under evaluation.

### 3.3.2.2

#### Improved data storage and downlink technologies

Commercially available data storage technology that will support capacities of up to 2000 gigabytes of digital video data is under investigation. In addition, possible data compression techniques, including JPEG, MPEG-II, and Wave-let technology, are being evaluated as techniques for real-time science operations.

### 3.3.2.3

#### Three-dimensional particle tracking

The need exists for a nonintrusive, three-dimensional, full-field velocity measurement device. This device would provide an improved method to measure three-dimensional fluid velocities quantitatively and simultaneously by mapping and tracking multiple tracer particles.

### 3.3.2.4

#### Nonintrusive digital pattern recognition and phase boundary locator

Data analysis would be much easier if a digital pattern recognition method could be used to analyze video images and calculate bubble/drop velocities. These calculations presently require tedious analysis.

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## A.4 FUNDAMENTAL PHYSICS

### A.4.1

#### Technologies

Following are brief descriptions of the technologies listed in section 3.4.1. Items with an asterisk were developed under the ATD Program. Items with a double asterisk were originally developed under the ATD Program and continued under the fundamental physics program.

### 3.4.1.1

#### Copper Ammonium Bromide (CAB) High-Resolution Thermometers (HRTs)

To perform the extremely precise temperature measurements required for the Lambda Point Experiment (LPE), the LPE team at Stanford University developed HRTs that can resolve temperature differences smaller than  $10^{-9}$  degrees. Along with the thermometers, an experimental platform capable of controlling helium samples to sub-nanokelvin stability was also developed. These thermometers, developed by the Micro-gravity Research Division, are now recognized by the scientific community as the state of the art in temperature measurement. Improved HRTs — those developed for the LPE — demonstrated higher noise levels in the space environment due to cosmic ray heating. In support of the Confined Helium Experiment (CHeX), which launched in July 1997 as part of the fourth United States Microgravity Payload (USMP-4) mission, HRTs were developed with faster response time to minimize this problem. These new thermometers also demonstrate improved sensitivity, resolving temperature differences below  $10^{-10}$  degrees. For the Critical Dynamics in Microgravity Experiment (DYNAMX), the copper elements of the HRT have been replaced with aluminum to reduce the cross section for cosmic rays. The DYNAMX team has developed HRTs with improved immunity to the degradation of performance due to cosmic rays that are a part of microgravity low-Earth orbit, essentially preserving the laboratory performance levels. This was achieved not only by replacing the copper elements with aluminum, but also by thermally insulating the sensing element from the cosmic ray heating of the surrounding niobium tube using Vespel™.

### 3.4.1.2\*\*

#### High-Resolution Capacitive Pressure Transducer

A simple way to construct and assemble a high-resolution capacitive pressure transducer has been developed at the Jet Propulsion Laboratory (JPL) under the ATD Program. This device is based on a capacitive readout technique and involves precision micromachining of silicon wafers. The transducer has demonstrated a resolution of 1 part in a billion at pressures in the 10-bar range. In the last year, a sealed design was developed to allow insertion of the pressure transducer directly into a liquid bath. A superconducting pressure gauge

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utilizing Superconducting Quantum Interference Device (SQUID) readout is being developed jointly by JPL and Stanford University. Recently, this device demonstrated a performance of better than 1 part in 10 billion across a pressure range from 1 to 10 bar. This technology will be packaged for the flight of the Superfluid Universality Experiment aboard the International Space Station (ISS) starting in fiscal year (FY) 1998.

#### **3.4.1.3\*\***

##### **Single-Electron Transistor (SET)**

The SET, initially developed at JPL as an ATD task, is an extremely sensitive detector of electric charge, with the potential to detect charge differences as small as  $10^{-6} e/\sqrt{\text{Hz}}$  ( $e$  is the charge on an electron), an improvement of over a million-fold beyond what other charge detectors can do. SETs are expected to find wide-scale application in high-resolution measurements of the future. For example, they could perform as readout amplifiers for capacitive gauges that can measure pressure, density, dielectric constant, or temperature. The SET will enable new kinds of measurements to be performed with very high resolution. Other very important applications for the SETs are as charge readouts for infrared-sensitive bolometers for use in astrophysics missions. Estimates of an improvement in the bolometer performance of approximately three orders of magnitude are predicted. Improved bolometer performance would enable researchers to probe space to unprecedented resolution. In FY 1997, a survey was made of the advances in this area, and new fabrication techniques were identified that will enable operation at higher temperatures.

#### **3.4.1.4\*\***

##### **High-Resolution Thermometry and Improved SQUID Readout**

The goal of this project at Goddard Space Flight Center is to advance the state of the art in high-resolution thermometers and SQUIDs. Key features of the thermometer are its fast response time and its potential immunity to cosmic ray effects on orbit. A special two-stage SQUID amplifier (TSA) that would be useful for ground applications, such as magnetometers for biological, geological, and chemical research, is also being investigated. The TSA is an improved version of the SQUID that cascades two stages of SQUID amplifier, using a single SQUID in the first stage and 100 SQUIDs in the second to obtain lower noise levels and wider dynamic range. Successful development of the TSA would enhance many of

the low-temperature and fundamental physics experiments proposed for Earth orbit.

#### **3.4.1.5**

##### **Prototype Miniature HRT**

A miniature HRT is under development using NASA Research Announcement (NRA) funds. A proof-of-concept device was fabricated, and preliminary tests have been performed. The device utilizes a samarium cobalt magnet to trap the initial magnetic flux in the circuit and  $\text{GdCl}_3$  salt pill technology for the thermometer itself. The main advantage of the new design is that it obviates the need for a massive electromagnet to do the initial field trapping. The HRT itself is also much reduced from the size used in previous flight experiments, offering the promise of improved designs to reduce the amount of heating experienced during launch of low-temperature experiments in the future. During FY 1997, sufficient progress was made so that a new ATD proposal was submitted and funded. This new ATD task will begin in FY 1998 with development of a new thermometer that is miniaturized and has improved sensitivity.

#### **3.4.1.6**

##### **Magnetic Levitator for Liquid Helium**

Researchers at Brown University have developed a magnetic levitator for liquid helium. They are using it to study drop dynamics of helium drops up to several millimeters in diameter under varying conditions.

#### **3.4.1.7\***

##### **Magnetostrictive Low-Temperature Actuators**

The objective of this project is to use the unique "giant" magnetostrictive properties of terbium, dysprosium, and zinc alloys as the prime mover in a series of actuators and mechanisms that include low-temperature valves, heat switches, precision positioners, and lead screwdrivers. These new materials have performance characteristics for low-temperature operation hitherto unavailable in any actuator. These materials and a family of devices are being developed for low-temperature use based on their unique properties of long stroke and high power with negligible energy dissipation. Several prototypes of these various devices were fabricated during FY 1997. New technology reports have been filed and various levels of intellectual property protection are being pursued. New types of these magnetostrictive materials have been developed, and methods for producing bulk polycrystalline rods are now being investigated.

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#### **3.4.1.8 Measurement of Ultralow Pressure at Low Temperature**

This work for the Satellite Test of the Equivalence Principle (STEP) project will demonstrate a pressure gauge that measures pressures as low as  $10^{-12}$  torr at temperatures between 2 K and 4 K. The technique is based on measurements of the relaxation time of an adsorbed helium film. The helium film will be measured with either a superconducting film bolometer or a quartz crystal microbalance. During FY 1997, frequency stability of  $2 \times 10^{-8}$  Hz was achieved.

#### **3.4.1.9\* Applications of Superconducting Cavities to Microgravity Research**

This ATD project has two main objectives: (1) the use of modern microwave electronics, high-quality factor (high-Q) low-temperature superconducting cavities, and high-resolution temperature control to develop an ultrastable oscillator system that will provide a comparison oscillator for the laser-cooled atomic oscillators now under development in the research program; and (2) to develop high-temperature superconductor (HTSC) materials, high-Q cavities, and electronics, and to integrate these components with a small cryocooler to provide an easy-to-use materials characterization system for use on the ISS. The ultrastable oscillator will strive for frequency stabilities better than  $df/f \cong 10^{-16}$  to provide comparison signals unattainable by other means. The HTSC materials characterization system will provide high-resolution sample characterization if the HTSC cavity obtains quality values greater than  $10^6$ . Both of these superconducting cavity systems will better benefit users on the ISS if they can be cooled with small cryocoolers that are capable of long-term operation in microgravity.

#### **3.4.1.10\* Development of an Electrostrictive Helium Valve**

Liquid helium is an excellent coolant for space apparatus that require cooling to below 10 K. So-called phase separators confine liquid helium to a storage tank. These separators “leak” into a vacuum chamber quantities of helium sufficient to provide the necessary cooling power. The proposed research is aimed at development of a new type of phase separator. This separator can also be used as a small, reliable cryovalve. The principle of the valve is based on the fact that the pressure of helium will

increase if an electric field is applied. This pressure increase will shift the superfluid transition of helium and, ultimately, will solidify it. If the high-field region is confined to a small aperture, then it will serve as a Josephson junction in moderate fields and as a valve when solid helium blocks it. Researchers at JPL manufactured submicron apertures, with electrodes to apply an electric field, and the apparatus for testing their behavior.

#### **3.4.1.11 Test Facility for Space Station–Era Cryoprobes**

A test facility for cryoprobes designed to fit in the Low-Temperature Microgravity Physics Facility (LTMPF), to be located in the Japanese Experiment Module Exposed Facility on the ISS, is being developed at JPL. The LTMPF, scheduled to be launched in November 2003, will accommodate two experiments at a time. Flights with new experiments are scheduled for every two years after the initial launch. The test facility will support technology development for the instruments to be flown on the LTMPF. It will include a vibration-resistant dewar to perform vibration tests at temperatures down to 1.5 K on flightlike cryoprobes; a flightlike cryoprobe built with the specifications of the current probe utilized in the LPE and in CHEX; and associated electronics, including accelerometers and data acquisition hardware and software. The cryoprobe will be equipped with an HRT similar to the ones flown on the LPE. This facility will allow tests for both launch-related and space station environment vibration levels. The flightlike cryoprobe will be used initially to quantify the susceptibility to launch load and low-level vibration heating, and in the future as a building block for the next generation cryoprobe. There is a great need for the miniaturization of the HRT subsystem, since space constraints on the space station facility will limit its physical size. The cryoprobe will be a test bed for the development of this new generation of HRTs.

### **A. 4. 2 New Technology Requirements**

Following are brief descriptions of the new technology requirements listed in section 3.4.2.

#### **3.4.2.1 Digital Signal Processing (DSP) SQUID technology**

While modern commercial SQUID systems display adequate resolution for the precise

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temperature measurements for which they are employed in this program, the SQUIDs are disrupted by electromagnetic interference (EMI) and have limited ability to track a fast-changing input signal. With the rapid application of fast digital processors to instrumentation, new SQUID systems are being built that employ DSP to amplify the signals generated by a direct current (DC) SQUID. The speed of DSP allows the output to track rapidly varying signals. These systems promise to simplify the employment of SQUIDs in the EMI-rife environments of the space shuttle and the ISS. A small effort to develop DSP SQUID systems showing better immunity to EMI and promising results similar to those demonstrated by the biomedical systems has been started at JPL and the University of New Mexico.

#### **3.4.2.2**

##### **Improved flight-quality radio frequency (RF) SQUID system**

SQUIDs are an integral part of the HRTs used in the LPE. The LPE SQUIDs will be reused on CHEX. Unfortunately, the company BTI, which manufactured these SQUIDs, no longer produces or supports this relatively old product. Additionally, many of the components used in the BTI SQUIDs are no longer available. This has left a void for future low-temperature experiments desiring use of SQUID technology. An effort is required to develop an available and supported commercial SQUID system for use in the space environment. The primary issues are EMI performance, thermal vacuum stability, and the ability to withstand the launch environment.

#### **3.4.2.3**

##### **Improved high-resolution pressure transducer**

The LPE, CHEX, and DYNAMX all have or will obtain data only along the saturated vapor curve where pressure above the liquid is maintained at the saturated vapor pressure of the phase diagram of liquid helium. During the brief periods of microgravity available for these shuttle-based experiments, this one set of pressure-temperature data is about all that can be managed. In the three- to six-month periods that will be afforded by the space station facility, investigators can explore other pressures, venturing well off the saturated vapor curve, to enrich the data return from their experiments. To maintain elevated pressures with stability comparable to the temperature control allowed by HRTs, improved pressure transducers must be developed.

This task would be aimed at developing such transducers utilizing superconducting and micromachining technologies.

#### **3.4.2.4**

##### **Vibration isolation for cryogenic systems**

Many experiments in low-temperature physics require the microgravity environment of space to enhance data return and to allow the study of phenomena not possible on Earth. It is the latter that make a vibration-isolated environment desirable. As the microgravity environment enables the study of new phenomena, it also introduces noise sources (i.e., vibrations) overwhelmed by other obstructions on Earth. Systems are desired that can suppress vibrations in the low-frequency range ( $>100$  Hz) by an order of magnitude or more.

#### **3.4.2.5**

##### **Precise thin-film deposition on cylindrical surfaces**

The STEP experiment will levitate hollow cylindrical masses around a cylindrical center shaft. Both the inner and outer cylindrical surfaces must have superconducting traces deposited precisely on their surfaces. In practice, a uniform film will be deposited on these surfaces, and then photolithographic techniques will be used to etch the precise pattern required. The printing of the circuit is very similar to the printing techniques used for xerography, so there are methods in place from which to begin development of the techniques necessary for this task.

#### **3.4.2.6**

##### **Long-lifetime cryogenics systems for the ISS**

As the nominal servicing interval for the ISS is about six months, and there are experiments in low temperature that need extended test periods, the technology necessary to take maximum advantage of the servicing interval while providing extended test periods is needed. This can involve the use of external refrigerators, which tend to be noisy, or some other mechanism to cool the outer shell of the system. Other potential technology developments, such as multilayer insulation or the use of novel materials for structural members, may be appropriate to the development of this system technology. The extension of lifetime realized in this task will lead to increased science return for each flight to the ISS.



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#### **3.4.2.7**

##### **Miniaturized HRTs**

Miniaturized HRTs enable the exploration of new avenues in science, such as spatially resolved phenomena that currently cannot be probed due to the confined space that is allocated to an instrument. An additional benefit of miniaturized HRTs is that they may also have inherently faster response times than the current paramagnetic salt HRTs. Some version of this size-reduced technology is essential to the concept of multiple experiments within one low-temperature instrument package, which will significantly reduce the average cost per experiment.

#### **3.4.2.8**

##### **Flight-qualified technology and instrumentation for laser cooling of atoms**

Because no experiments in the area of laser cooling have been conducted in spaceflight, no flight hardware has been developed. Much of what is needed for these experiments, however, has been developed and flown as part of other experiments. This task would develop experimental apparatus for laser cooling research that will survive the rigors of launch and will operate well in the environment of the ISS.

#### **3.4.2.9**

##### **Visual-access cryogenic system or cryogenic high-speed video camera**

Four of the ground-based investigations now being pursued in the low-temperature microgravity physics program require visual access to the experimental cell to obtain data. Such access is obtained routinely in ground-based dewars and cryoprobes, but the present flight facility and flight probes do not allow visual monitoring. It seems likely that the access can be managed by lines of sight parallel to the dewar cylinder axis, so only the cryoprobe insert, not the facility, need be modified. Visual access in such experiments could also be provided by a high-speed video camera able to operate at cryogenic temperatures. This task would explore concepts to permit visual monitoring of events in the experiment cell.

#### **3.4.2.10**

##### **Flight-quality DC SQUID system**

The development of SQUIDs started with low-frequency RF systems, which were changed to higher frequency systems to improve resolution, and then were transformed to DC SQUIDs with yet

higher resolution and lower noise. The commercial market for SQUIDs is now moving to the simpler, lower-noise DC SQUID technology. The LPE began during the earliest period of SQUID development, and CHEX also used low-frequency RF technology in its 1997 flight. However, lowering the noise of the SQUID permits shrinking the size of the sensing element with no loss of signal to noise. The adaptation of commercial DC SQUIDs for flight will simplify the miniaturization task, with its enabling of new measurements and simplifying of the launch configuration. Perhaps more significantly, eventually the DC technology will be the only one supported by industry, so replacement parts will likely become unavailable for the RF SQUIDs.

#### **3.4.2.11**

##### **Flight-quality DSP SQUIDs**

The DSP SQUID systems that are described in 3.4.2.1 would, under this task, be built to operate well in the thermal, vacuum, and EMI environments of low-Earth orbit, and to survive launch. A DSP chip has been flown on other missions, so this task must be sure to employ one that has demonstrated immunity to cosmic radiation. This task would essentially take the DSP SQUID system developed under 3.4.2.1 from a ground system to a flight-hardened system and demonstrate flightworthiness by environmental testing.

#### **3.4.2.12**

##### **Ultrahigh-vacuum production and measurement techniques**

The production of ultrahigh vacuum, reaching pressures below  $10^{-9}$  torr, will benefit more than one type of low-temperature experiment. The calorimetric experiments, like those presently being flown on shuttle flights, require isolation from the surrounding environment to minimize heat leaks so that temperature drifts are small; pressures in the  $10^{-9}$ – $10^{-10}$  torr range are desirable. During the STEP experiment no convective forces can disturb the levitated masses, which means pressures below  $10^{-11}$  torr are required. This task would use sorption pumping and specially developed pressure measurement techniques to demonstrate that such pressures can indeed be obtained.

#### **3.4.2.13**

##### **Multiple experiment platform for low-temperature physics experiments**

Development of a multiple experiment platform envisioned for low-temperature experiments would



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be an extension of the current technology and would ultimately lower the cost per experiment, since the cost of a given launch would be apportioned over two or more experiments. This technology would also increase the access to space for low temperature scientists, as it is currently limited by the number of launches available and the resources available to support a launch. The new experiment platform for the space station should also minimize the amount of vibration heating experienced during launch, with the ultimate goal of obviating the need to use exchange gas during launch as thermal protection.

#### **3.4.2.14**

##### **Low-noise cryocoolers**

Low-noise cryocoolers could be a source of a cryogenic environment alternative to the stored cryogen systems presently available. The primary shortcoming of today's cryocoolers is that they are significant sources of vibration. This vibration energy would perturb the types of high-resolution experiments performed at low temperatures to the extent that the data obtained would be dominated by the noise source. A hybrid system consisting of a low-noise cryocooler and a stored cryogen system could provide a low-temperature, low-noise environment in space for many months.

#### **3.4.2.15**

##### **Flight-quality dilution refrigerator**

The forefront of ground-based experimentation in condensed matter physics has moved steadily to lower temperatures over the past several decades, now reaching the range near 10 microdegrees above absolute zero. The method of choice for obtaining temperatures below 1 K is the dilution refrigerator, which permits temperatures below 0.01 K to be maintained for several months. As presently configured, dilution refrigerators employ several phase boundaries that require gravity for their stable location. New concepts have been developed to get around these interface problems, and one is now being explored at JPL with seed money from the Director's Discretionary Fund. At least one experiment that requires these low temperatures was proposed in response to the fundamental physics NRA. The combination of ultralow temperatures and a low-gravity environment will provide investigators a new realm of experimental parameters to explore.

#### **3.4.2.16**

##### **Flight-quality GdCl<sub>3</sub> thermometer**

GdCl<sub>3</sub> thermometers are extensions of the current CAB paramagnetic salt thermometers, the difference being the salt utilized. GdCl<sub>3</sub> offers the potential of higher sensitivity in the vicinity of the lambda transition of helium and especially at the higher temperatures of the liquid-vapor critical points of <sup>3</sup>He (3.2 K) and <sup>4</sup>He (5.4 K). GdCl<sub>3</sub> is also a stable material to work with, simplifying the implementation of the HRT. The further development of GdCl<sub>3</sub> HRTs can yield higher resolution than the ~10<sup>-10</sup> K available today from CAB HRTs, so these new devices could improve the science return from low-temperature experiments.

#### **3.4.2.17**

##### **Flight-quality laser-cooled atom trap facility**

The interference of multiple laser beams in a region where an atomic beam of laser-cooled atoms exists can generate areas of force on the atoms. These areas have a pattern of minima and maxima. The valleys and peaks can be used to trap the cold atoms for significant times so that they can be studied. For example, their quantum states can be studied by causing transitions between two states and observing the emitted or absorbed radiation. The slowed motion of the cold atoms enhances the precision of such measurements; however, there is a drain of atoms from the trap due to gravity. No flight hardware exists to permit such experiments to be conducted in space in order to avoid the gravitational drain. This task would develop the flight hardware for these experiments.

#### **3.4.2.18**

##### **Flight-qualified <sup>3</sup>He refrigerator**

The present facility for low-temperature condensed matter experiments provides a liquid helium bath cooled to temperatures just below 1.8 K, which is fine for experiments that explore the lambda transition at 2.177 K or phenomena at higher temperatures. However, there are a wealth of new phenomena to explore below 1 K. A <sup>3</sup>He refrigerator would allow experiments to be cooled to below 0.5 K. Several of the presently funded ground-based studies will require subkelvin temperatures for their experiments. A <sup>3</sup>He refrigerator would provide such temperatures in the simplest manner. A <sup>3</sup>He refrigerator has been developed for space use on an infrared mission recently flown by the Japanese, so this task only requires the adaptation of that technique to the program's heat-load requirements and to the geometric constraints.

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#### **3.4.2.19**

##### **Accelerometers for use at helium temperatures**

Accelerometers for use at helium temperatures would help experimenters in the post-processing of their data by providing a characterization of the acceleration environment at the helium test cell. This characterization would improve the overall quality of the science result by minimizing the error band due to a significant noise source, such as extraneous accelerations. The warm accelerometers used by the Space Acceleration Measurement System require interpolation of the data from the warm location where the data are taken to that point where the test cell is located in the cryostat. Cold accelerometers can be located much closer to the experimental cell, where the accelerations they measure will much more accurately represent the accelerations experienced at the cell.

#### **3.4.2.20**

##### **Fast-response high-resolution thermometry**

Fast-response high-resolution thermometry would enable experimentation in some new areas of physics, particularly those areas where it is desirable to study transient phenomena when the relaxation time is short. High-speed HRTs would also have the effect of mitigating cosmic ray noise, as these data anomalies would be filtered out in post-processing. Alternatively, a high-speed HRT could be used in the study of the cosmic ray environment on orbit. These thermometers still require  $10^{-10}$  K or better resolution, but sampling rates of 100 Hz or more would be enabled. If the present task to develop smaller, faster thermometers does not succeed for lack of sensitivity, other methods to increase the response speed of the HRTs should be explored.

#### **3.4.2.21**

##### **Miniature high-reliability low-temperature valve**

All low-temperature experiments have a need for highly reliable cryogenic shut-off valves in various forms. In the space station era, the extended experimentation time afforded by the longer cryogenic lifetime will enable experiments to be done at various pressures. Therefore, valves are needed that can be operated repeatedly in the microgravity environment in a reliable manner in order to adjust the sample pressure under investigation.

#### **3.4.2.22**

##### **Flight-quality ultrastable frequency standard**

Many experiments in the atomic physics and relativistic and gravitational physics areas rely on

highly accurate clocks for successful performance. To allow researchers in these fields to make measurements in the microgravity environment of space, flight-qualified clocks will be needed.

#### **3.4.2.23**

##### **Low-noise cryogenic instrumentation amplifiers**

All cryogenic experiments proposed for NASA missions depend on precise measurements made by electrical sensors located near an experiment cell. The cables between the sensor and the warm read-out electronics are often highly susceptible to electrical noise. A low-noise cryogenic flight-quality instrumentation amplifier located adjacent to the sensors would amplify the signal before noise is introduced by the cabling.

#### **3.4.2.24**

##### **Telescience flight software tools**

Software controlling microgravity flight experiments often does not present an efficient, simple interface to the investigator. Each flight project requires a great deal of effort to be expended on writing and debugging real-time control software. Longer run times for experiments aboard the ISS will greatly increase operating costs unless the investigators can control their experiments directly through simple, reliable remote interfaces. Ground-based experimenters have solved these problems with graphic, real-time programming languages (e.g., Labview). The reliability, response time, and hardware compatibility of these tools must be improved before they can be used in real-time flight experiments.

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## **A.5 MATERIALS SCIENCE**

### **A.5.1**

#### **Technologies**

Following are brief descriptions of the technologies listed in section 3.5.1. Items with an asterisk were developed under the ATD Program.

#### **3.5.1.1**

##### **Coupled Growth in Hypermonotectics**

The objective of this investigation is to gain an improved understanding of solidification processes in immiscible alloy systems. A portion of this study involves the development of experimental techniques that will permit steady-state coupled growth of hypermonotectic composition samples to produce

aligned microstructures. A parallel effort is under way to develop a model for the coupled growth process in monotectic systems. This analysis starts with the basic equations for diffusion-controlled growth and avoids many of the simplifying assumptions often utilized in similar analyses. Great strides have been made in the modeling area with the inclusion of several factors that specifically address behavior in immiscible systems. As part of this study, three microgravity directional solidification experiments in immiscible alloys were carried out aboard the Life and Microgravity Spacelab (LMS) mission in 1996. Special aluminum nitride ampoule assemblies that used a piston-and-spring arrangement to accommodate shrinkage were utilized in these experiments. The results obtained from these initial experiments will be used to test the model and to upgrade the ampoule assemblies for future investigations planned for the ISS. A microgravity glovebox investigation is currently under way to study the wetting characteristics of immiscibles as part of the overall project. Experimentation occurred aboard USMP-4, which was launched in November 1997. The results obtained from these experiments will aid dramatically in understanding solidification processes in these intriguing systems. These systems show great potential for use in many engineering applications.

### **3.5.1.2**

#### **Orbital Processing of High-Quality Cadmium Telluride**

The modified seeded Bridgman-Stockbarger technique was utilized in the Crystal Growth Furnace (CGF) to grow cadmium zinc telluride crystals in microgravity. The reduction of buoyant convection increased chemical homogeneity, and the lack of hydrostatic pressure enabled a significant reduction in defect density. Improved crystals can be grown for use in the fabrication of medium- and long-wavelength infrared sensors and beta and gamma ray nuclear detectors. Experimental results showed consistency with high-fidelity thermal and thermo-mechanical stress models.

### **3.5.1.3**

#### **Growth of Solid-Solution Single Crystals**

Improved crystal growth methods focusing on the melt, telluride-solvent growth, and growth in magnetic fields are under development. The Advanced Automated Directional Solidification Furnace (AADSf) was used to grow a 16-cm-long  $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$  alloy crystal. Orbital and residual

acceleration effects were correlated to various crystal features and alloy composition changes. Application of magnetic fields was shown to reduce radial composition variations in the crystals, agreeing well with theoretical predictions. A second experiment will be conducted in the AADSf to verify and improve the information previously obtained. The electrical and optical properties of these materials make them important to a wide range of technological applications in the areas of sensors and lasers with applications to optical computing, communications, and national defense.

### **3.5.1.4**

#### **Crystal Growth of II-IV Semiconducting Alloys by Directional Solidification**

A new seeded method has been developed for the growth of mercury zinc telluride crystal ingots from pseudobinary melts using the Bridgman-Stockbarger directional solidification technique in the CGF. Supporting normal-gravity studies were conducted in the Ground Control Experiments Laboratory. A vapor transport method was developed to grow 2-cm zinc telluride seed crystals in fused silicon ampoules. The effect of reduced gravity on the crystal growth of mercury zinc telluride and mercury zinc selenide is sought, particularly on the fluid dynamic and compositional redistribution phenomena, which occur during the crystal growth of solid-solution semiconducting alloys that have large separation between the liquidus and solidus of the constitutional phase diagrams. Researchers are seeking more accurate control of properties for materials that are important to electronics and infrared detectors.

### **3.5.1.5**

#### **The Study of Dopant Segregation Behavior During the Growth of GaAs in Microgravity**

Sample/ampoule design configuration was completed for use in the CGF to fabricate and process selenium-doped GaAs crystals. The crystals will be characterized by optical, electrical, and chemical properties. Results will be compared with theoretical predictions.

### **3.5.1.6**

#### **Temperature Dependence of Diffusivities in Liquid Metals**

The technological goals of the Self-Diffusion in Liquid Elements (SDLE) project are to develop a methodology for the measurement of the self-diffusivity of various liquid elements over a

wide temperature range. The diffusivities are determined *in situ* utilizing radioactive tracers. This *in-situ* method has the desirable feature of enabling the measurement of diffusivities at several temperatures with a single sample. Thus, this methodology requires fewer samples and less flight time than traditional approaches. The SDLE scientific goals are to accurately measure diffusion coefficients of several elements at multiple temperatures to determine if there is class-like behavior in these materials, and if so, to determine the correct mathematical description of that behavior. The Liquid Metal Diffusion (LMD) experiment was selected as a risk-mitigation precursor to the SDLE. It utilized the Canadian vibration isolation system called the Microgravity Isolation Mount (MIM), located on the Russian space station, *Mir*, from January through March 1997. Indium metal samples were processed with the MIM in two different modes, one providing active isolation, one inactive. Goals of the LMD experiment were to test the diffusion measurement technique at a single temperature using indium and to determine how easily these diffusion coefficient measurements are contaminated by convection arising from g-jitter. It is important to note that while the LMD investigation was justified by the mitigation of risk for the SDLE project, it also had the additional scientific goal to determine the diffusion coefficient of indium at 200° C with greater accuracy than had been previously attained. Analysis and conclusions from the experiment data have not yet been published.

#### 3.5.1.7

##### **Particle Engulfment and Pushing by Solidifying Interfaces**

Techniques were developed to study the pushing and engulfment of particles by planar liquid/solid interfaces during solidification of metallic alloys. A new, nondestructive technique was developed to characterize particle positioning before and after processing. The method of testing samples was validated in the Advanced Gradient Heating Facility furnace in France.

#### 3.5.1.8

##### **Crystal Growth of ZnSe and Related Ternary Compound Semiconductors by Physical Vapor Transport**

A novel vapor transport three-zone heater translating method was developed, and optimized methods of growing ZnSe and related ternary

compounds were sought. Optical *in-situ* monitoring techniques to study the growth kinetics and vapor transport process were investigated, and various characterization techniques are being perfected and standardized. Analytical and theoretical methods were being developed to evaluate crystals. Mass flux was measured and compared to theoretical calculations. Horizontal and vertical growth of ZnSe, ZnSeTe, and ZnSeS was performed and the crystals characterized. These materials are useful for opto-electronic applications, such as highly efficient light-emitting diodes and room-temperature lasers in the blue-green region of the visible spectrum.

#### 3.5.1.9

##### **Test of Magnetic Damping of Convective Flows in Microgravity**

Experimental and numerical methods were developed to investigate the use of magnetic damping to suppress convective flows driven by gravity, vibration, or surface tension gradients. Materials to be studied are a dilute alloy (Ga-doped Ge) and three solid solutions (Ge-Si, InSb-GaSb, and Cu-Ni) with two diameters using the Bridgman method. Diffusion-controlled growth was accomplished, and the accuracy of numerical methods for predicting the needed magnetic field was validated. Sample containers and demarcation techniques were also developed.

#### 3.5.1.10\*

##### **Real-Time X-Ray Microscopy for Solidification Processing**

An X-Ray Transmission Microscope (XTM) for the *in-situ* and real-time observation of interfacial processes in metallic systems during freezing or solid-solid transformation is being developed. The XTM will provide a resolution for specimen features of 10–100  $\mu\text{m}$ , at rates of 0.1 to 20  $\mu\text{m}/\text{sec}$ , at temperatures of 1100° C, and gradients up to 50° C/cm. Other features include contrast sensitivities sufficient to detect a 2–5% difference in absorbance; one, two, or four exposure times of a few seconds in duration; and the ability to record stereo pairs for depth information. Physical processes that play a role in the determination of technologically important properties of solids can be studied in opaque materials, especially metals, with the XTM. Specifically, studies of interfacial morphologies and particle-interface interactions can be observed.



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### 3.5.1.11

#### **Physical Properties and Processing of Undercooled Metallic Glass-Forming Liquids**

The project objectives include: (1) the development and implementation of noncontact alternating current (AC) modulation calorimetry (ACMC) for measuring the specific heat and thermal conductivity of reactive liquids during electrostatic levitation (ESL) at elevated temperatures; and (2) the development of atomic diffusion measurements in liquid alloys during containerless processing in ESL. Software was written to produce AC modulation of the basic laser power. This allows for the implementation of the ACMC method. The second part of the project involves developing methods for measuring atomic diffusion in the liquid state using the High-Temperature Electrostatic Levitator. In this area, preliminary ground-based atomic diffusion experiments on liquid samples contained in graphite crucibles have been carried out. The samples are situated in a high-vacuum chamber and can be heated by induction heating. Direct measurement of the sample temperature can be made with thermocouples embedded in the crucible. Active feedback control of the power enables rapid heating and subjects the samples to a well-defined temperature history. In this facility, preliminary experiments were carried out using concentric samples of cylindrical symmetry having a concentration difference in the two regions. The entire sample is subjected to thermal treatment in the liquid state for controlled times to produce a chemical interdiffusion profile. The samples are then solidified to the glassy state, and diffusion profiles are analyzed using scanning Auger analysis of the cross-sectioned samples.

### 3.5.1.12

#### **Investigation of the Relationship Between Undercooling and Solidification Velocity**

A model to correctly predict solidification velocity as a function of undercooling is being developed. Numerical, microstructural, and surface analysis, as well as orientation imaging microscopy, are used in the development of this model, which has four regimes based on the amount of undercooling. The first regime occurs for low undercoolings in which the solidification velocity increases slowly as a function of undercooling. The second regime marks the beginning of nonequilibrium processes occurring at the solid/liquid interface. A third regime occurs in situations in which the growth rate is predominantly

controlled by the thermal driving force for solidification. The fourth regime begins when the interdendritic spacing is small enough to allow the thermal and solutal fields to interact. In addition, a model has been developed for pure materials and materials with low solute concentrations. For this model, there are two observed regimes for the solidification velocity as a function of undercooling. In the first regime, the solidification velocity at low undercoolings is thermally controlled and the dendrites are widely spaced. In the second regime, the dendrite spacings are small and solutal and thermal fields begin to overlap. As in the model for higher-concentration alloys and intermetallics, the energetics of the interface are controlled by the competition between the driving force for solidification, the activation barrier to solidification, and the energy consumed by the presence of solute at the interface. In both models, the high undercooling regime is in disagreement with currently accepted theories. The current models predict collision-limited growth to define the upper limit of solidification velocity, which means that the maximum velocity is on the order of the speed of sound in the liquid. The maximum velocities measured in this study are approximately one-fourth of the values predicted by collision-limited growth.

### 3.5.1.13

#### **Space- and Ground-Based Crystal Growth Using a Magnetically Coupled Baffle**

Experimental and numerical methods were developed to investigate the use of submerged, disk-shaped baffles to control heat and mass transfer during semiconductor crystal growth in vertical Bridgman configurations. Ga-doped Ge, GaSb, InSb, and GaInSb crystals were grown both with and without a submerged baffle present in the melt. Numerical calculations indicate that the use of a baffle reduces the flow velocities in the melt by a factor of approximately 15. This magnitude of a reduction of natural convection can lead to significant improvements in segregation.

### 3.5.1.14

#### **Compound Semiconductor Crystal Growth in a Microgravity Environment**

The primary purpose of this investigation is to determine how gravity-driven convection affects the composition of the PbSnTe alloy, which is subject to convection induced by both thermal and compositional differences. The AADSF was used to grow three totally separate crystals by segmenting the

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ampoule. The experimental variable for the three cells was the direction of the microgravity. The shuttle was flown in three different growth directions used by different crystal growth systems on Earth. The results of this experiment were totally unexpected. The crystals started to grow as intact solids; then, after approximately one centimeter of growth, they necked down almost to cut-off. The diameter then increased to fill the ampoule except for large pores and surface channels that ran almost the remaining length of the crystal. The existence of pores during the growth period presented the conditions for surface tension-driven convection, and the compositional analysis subsequently showed the effects of convection during the growth process. Possible causes of the void formation were addressed in an experiment conducted in the AADSF during USMP-4.

#### **3.5.1.15**

##### **Orbital Processing of Eutectic Rod-Like Arrays**

The objective of this program is to utilize the orbital microgravity environment to conduct unique experiments to evaluate process models that describe convective influences on the solidification of regular low volume-fraction eutectic alloys. This program will directionally solidify low volume-fraction, regular eutectic structures in orbit, under diffusion-controlled growth conditions. Complementary *in-situ* diagnostics will be applied to monitor and/or perturb the solidification interface velocity and to precisely monitor the solidification interface temperature (undercooling) and shape. Postflight analyses will quantitatively determine the relations between the solidification interface velocity, solidification interface temperature (undercooling), and microstructural parameters (rod diameter, inter-rod spacing, and volume fraction). Identical experiments will be conducted terrestrially under damped (gravitational stabilization and applied magnetic fields) and undamped conditions to provide a quantitative 1 g baseline for comparison.

#### **3.5.1.16**

##### **Interface Pattern Selection Criterion for Cellular Structures in Directional Solidification**

This research is aimed at establishing the fundamental physics that govern the selection of unique microstructural features under a given growth condition. The fundamental role of transport and interfacial energy will be quantitatively evaluated through the development of a rigorous quantitative model for diffusion-controlled growth. This model

will be tested in a microgravity environment in which convection effects are minimized. Initial ground-based studies will be carried out in Al-4 wt% Cu and Al-15 wt% Cu alloys to establish the range of conditions over which cellular structures are stable. The condition for cellular to dendritic transition will also be established. Within the cellular range, the steady-state cellular structure will be characterized with respect to tip temperature, tip composition, cell amplitude, three-dimensional cell shape, and microsegregation patterns. The above characteristics will be examined under different velocities and temperature gradients. Experiments will also be carried out in a Pb-Sn system, which requires low furnace temperatures. A new furnace facility design was developed that can allow several samples to be solidified simultaneously under the same growth conditions and under highly controlled thermal conditions.

#### **3.5.1.17**

##### **Comparison of Structure and Segregation in Alloys Directionally Solidified in Terrestrial and Microgravity Environments**

This is a program to conduct experiments in a long-duration microgravity environment with the objective of studying dendritic microstructures and segregation in directionally solidified dendritic alloys. The research builds on a previous grant in which an extensive database was generated on directionally solidified Pb-Sn alloys and simulated solidification of the alloys using continuum theory of porous media. Directional solidification experiments on a Pb-Sn alloy and on a Al-Cu alloy have been outlined using different growth conditions to affect different dendritic microstructures. Numerical modeling to simulate transport phenomena and solidification in the planned experiments is currently under way. Preliminary experiments related to capsule materials are in progress, and as ingot samples are prepared, they will be characterized for dendrite morphology, macrosegregation, and possibly for microsegregation.

#### **3.5.1.18\***

##### **Advanced Heat Pipe Technology for Furnace Element Design**

Heat pipe technology has proven to be of use as an isothermal liner. The goals of this project are to fabricate liners to operate at up to 1500° C; determine the feasibility and establish the protocol for the incorporation of liquid metal heat pipes as furnace liners in a human-tended space environment; and develop a furnace with no moving parts to solidify



or cool materials with a high degree of control. The performance of the device, the Moving-Gradient Heat Pipe Furnace (MGHPF), will be extensively characterized. The first three heat pipes were designed and fabricated, the MGHPF was installed at Marshall Space Flight Center, and initial testing has begun. Crystals of several materials were grown. The remainder of the program in FY 1997 was devoted to extensively testing the temperature uniformity of the inner surface of the heat pipe, determining the longevity of the heat pipe, and analyzing potential failure modes.

#### **3.5.1.19\***

##### **Ceramic Cartridges via Sintering and Vacuum Plasma**

A manufacturing process is under development for containment cartridges used in high-temperature (1200–2000° C) CGFs. A thermal spray process has been used to build up refractory metals and ceramics into a containment cartridge for high-temperature single-crystal semiconductor growth experiments. These plasma spray-formed materials have been evaluated for mechanical properties, density, microstructure, and resistance to liquid metal attack. Forming techniques and the resultant mechanical and metallurgical properties have been identified. In FY1997, parameter development and deposit characterization have been completed for a variety of refractory materials, including tungsten, molybdenum, tantalum, niobium, rhenium, alumina, and aluminum nitride/zircon. This work has been expanded to document the chemical reactivity/compatibility of 14 semiconductors or other crystalline materials with five refractory cartridge materials.

#### **A.5.2**

##### **New Technology Requirement**

Following is a brief description of the new technology requirement listed in section 3.5.2.

#### **3.5.2.1**

##### **Development of an optical characterization facility for D-X defects in bismuth silicon oxide (BSO)**

A characterization technique is needed for the technologically important nonlinear optical material BSO. This work is an important component of the NASA effort to establish a role for microgravity research in new areas of materials science. Development of an effective technique to characterize crystal quality in BSO will be essential in establishing a program of space research in crystal growth of

nonlinear optical materials. Because nonlinear optical materials are projected to have a major role in the future of computing and communications, development of a research program in this area is of strong programmatic interest.

## **A.6 MULTIDISCIPLINE TECHNOLOGIES**

#### **A.6.1**

##### **Technologies**

Following are brief descriptions of the technologies listed in section 3.6.1. Items with an asterisk were developed under the ATD Program.

#### **3.6.1.1\***

##### **Free-Float Trajectory Management**

An instrument was designed that takes information from accelerometers and aircraft control inputs, such as wind speed, altitude, and control surface deflections, and displays it to pilots in such a graphic and immediate manner that the pilots can use the display to guide reduced-gravity aircraft to attain extended periods of free float. The pilot sees a computed package position inside the fuselage and responds by controlling the aircraft to keep the package free-floating.

#### **3.6.1.2\***

##### **Vibration Isolation and Control System for Small Microgravity Payloads**

Many microgravity science experiments will require an active isolation device to provide a quiescent acceleration environment on the space station. In this ATD effort, the technology developed for and demonstrated by the Suppression of Transient Accelerations by Levitation system will be extended for a minimum-volume vibration isolation system for the space station. Unique to this device will be modularity and small size, which are necessary for rapid turnaround and low cost for multiple users. Since the system will be application-independent, there will be no additional cost for accommodating different users. In addition, this system provides the unique capability of measuring the absolute acceleration, independent of accelerometers, as a by-product of the control system. It will also have the capability of generating pristine accelerations to enhance experiment operations. An umbilical follower device will be developed to reduce the power needed by the isolation device and give greater utility to the actuator for use with larger

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systems such as furnaces. During FY 1997, significant progress was made in regard to the unique self-sensing position measurement concept for which a patent application has been submitted. In addition, a conceptual design was developed for the prototype six-degrees-of-freedom isolator for the Microgravity Science Glovebox.

#### A.6.2 New Technology Requirement

Following is a brief description of the new technology requirement listed in section 3.6.2.

##### **3.6.2.1 Miniature microscope**

Both protein crystal growth and colloid physics experiments could benefit from a microscope small enough to add to experiment apparatus. This would allow direct observation of ordering in colloids, supplementing laser light scattering and Bragg scattering techniques. If small enough, several such microscopes could be placed on adjacent protein crystal growth cells, permitting observation while not inducing g-jitter.

# Appendix B

## FY 1997 Advanced Technology Development Program and Project Descriptions

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### B.1 PROGRAM DESCRIPTION

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Experience with microgravity research has shown that technology limitations generally emerge during experiment development and hardware design. In order to address these limitations, the Microgravity Research Division has implemented the ATD Program.

The ATD Program's main goal is to develop technology that will enable new types of scientific investigations and advance current research. This is accomplished by enhancing the capability and quality of current experimental hardware and by overcoming existing technology-based limitations identified in the Microgravity Research Program.

The ATD Program anticipates the needs of microgravity researchers and provides for the development of dynamic, state-of-the-art technology before it is needed on the critical paths of specific flight programs. This research already has provided numerous advances and breakthroughs in experiment techniques, sensor technology, and precise measurement ability. ATD projects are funded through an initial demonstration of feasibility and suitability for use in either the ground- or flight-based programs.

ATD Program application and acceptance is a two-step review process. Requests for more information should be directed to the address below:

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### B.2 PROJECT DESCRIPTIONS

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The following pages contain descriptions of projects in the FY 1997 Advanced Technology Development Program. A complete description of the program, including information on the development of project proposals, the selection of concept papers, the selection and approval of new ATD projects, and annual progress reports, can be found in the document *Advanced Technology Development Program 1997 Annual Update* (NP-1998-01-02-MSFC) and on the World Wide Web at <http://mgnwww.larc.nasa.gov/ATD97/>.

# Magnetostrictive Low-Temperature Actuators

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## OBJECTIVES

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A single advance in materials science can lead to rapid advancement in the related technologies of application. The invention of giant magnetostriction at the Naval Surface Warfare Center is such an advance. Long-throw, high-force actuation at cryogenic temperatures, which was previously difficult, is now much easier. A second advance in materials science that impacts the application of magnetostriction is a radical reduction in the size and an increase in the current-carrying capabilities of high-temperature superconductive solenoids.

These two factors work synergistically in generating mechanical movement and producing force at cryogenic temperatures. Consequently, the potential exists for the quick advancement of related techniques for producing low-temperature actuators and mechanisms. This ATD project was created to explore and exploit this anticipated “solution-rich” area of material development and application. The objectives of the project are to better understand these advances in materials science; to learn more about their implications for lower cost and more reliable actuator design; and to realize this inherent potential in the production of a series of prototype valves, heat switches, and motors, based on the magnetostrictive approach.

## METHOD

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The basic method used to develop low-temperature magnetostrictive materials in this ATD project has three separate elements. The first element is a collaboration with the Department of Material Science at the California Institute of Technology (Caltech). Because development of the science of magnetostriction is important to the success of the project, a program of cooperative work in magnetostrictive science was established using funding from both this ATD project and Caltech. This effort continues to be productive in shaping the understanding of the physics of magnetostriction and its applications.

The second element of the approach is based on the need to reduce the cost and increase the uniformity of the magnetostrictive crystals that are available for use by principal investigators funded by the Microgravity Research Program. Conducting research in manufacturing methodology for these

materials should lead to more cost-effective and higher-quality crystals, which will, in turn, lead to greater potential applicability of these materials.

Applications development is the third element on which this project is focused. A program of application and inventions has been implemented, resulting in products that were the subject of nine New Technology Reports (NTRs) this year. A program of end-user education and market cultivation has also been a significant part of this project. This program includes collaboration with several small companies that have joined efforts in high-temperature superconductor applications with magnetostriction and mechanisms development. Future tasks that could arise from this collaboration include the development of acoustic pumps for microgravity cryogen transfer and of magnetometers that use magnetostrictive crystals as the primary sensor element and fiber optics for readout.

## SCHEDULE & MILESTONES

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Many of the identified goals of the original four-year proposal have been achieved ahead of schedule. A working valve has been produced along with functional prototypes (albeit of a preliminary type) of all of the types of devices except for the heat switch. The heat switch delivery was pushed back in a minor restructuring of the schedule.

The applications and devices effort has been particularly productive, with eight NTRs for devices submitted in the last year. A ninth NTR was submitted for a new magnetostrictive material to be used in low-temperature applications. This material is easier to produce and lower in cost than present materials used for such purposes. The NTRs are being reviewed at the time of this writing with regard to their commercial viability and their potential for meaningful patent protection.

## APPLICATION & TECHNOLOGY TRANSFER

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Two versions of magnetostrictive valves have been constructed from the serial number 001 chassis, the second of which is in use on a ground-based experiment related to the Microgravity Investigation of Scaling Theory Experiment. The serial number 002 chassis is currently in production. In a joint

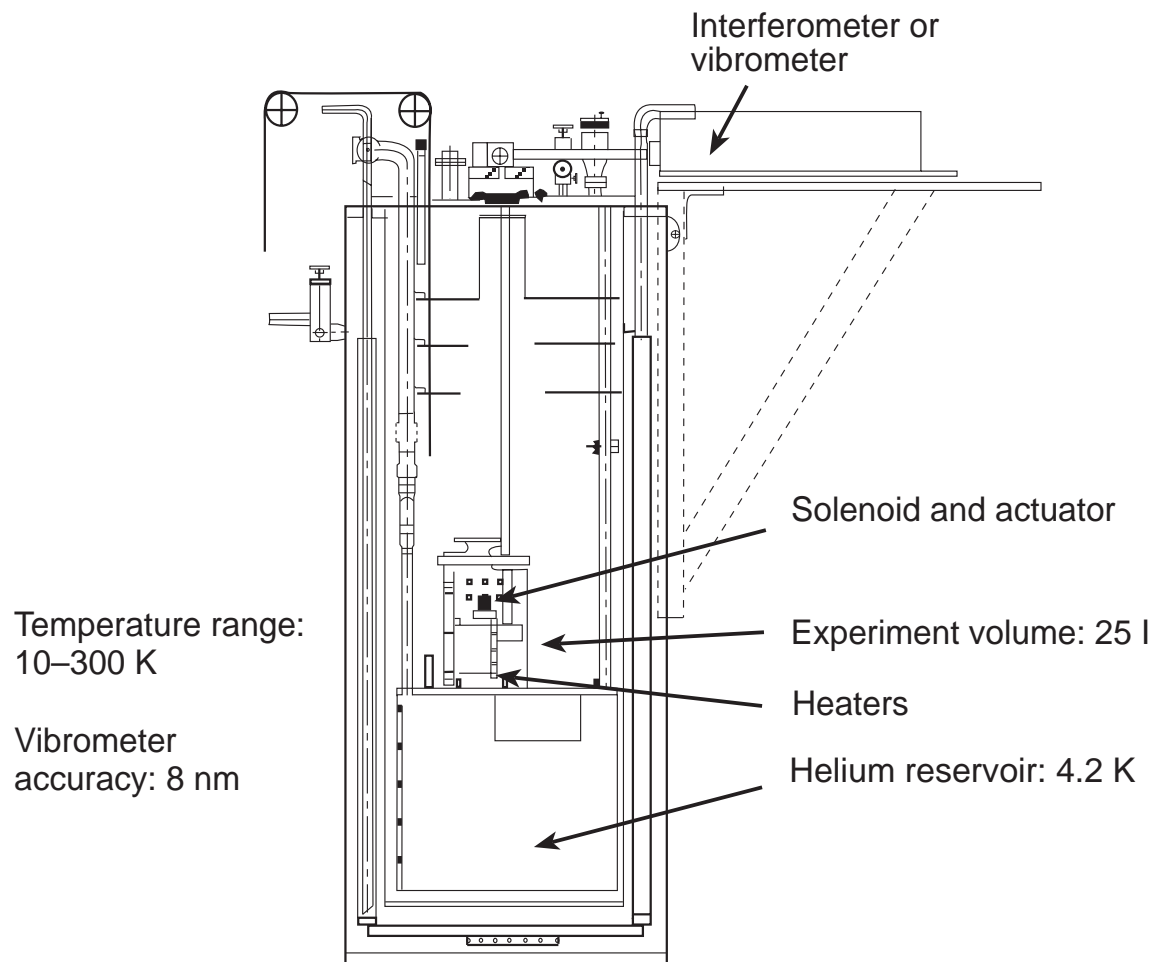


Figure 1: Dilatometer for cryogenic mechanism test; liquid helium probe and dewar schematic

technology-development cooperation agreement, American Superconductor furnished a high-temperature superconductor that was used to successfully drive the valve at liquid nitrogen temperature. The valve is liquid helium-tight after many cycles.

Detailed concept development of the heat switch is complete, as are some piece-part drawings.

Working prototypes of linear actuators have been developed and tested at Cornell University, and Grumman Aircraft, Inc., has proposed to use one in an etalon. Etrema, Inc., will submit a Small Business Innovative Research (SBIR) proposal based on some of this technology.

A prototype of the rotary actuator has been tested at 77 K with good results. The device was also used as a drive element in a threadless lead screw in the Next Generation Space Telescope. A resolution of 10 nm was achieved.

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# Advanced Heat Pipe Technology for Furnace Element Design in Spaceflight Applications

## OBJECTIVES

This project focuses on two potentially important advances in heat pipe and furnace technology. The first is the development of a heat pipe that will operate as an isothermal furnace liner capable of processing materials at temperatures up to 1500° C. The isothermality and control associated with a heat pipe greatly exceeds that possible in traditional wire-wound furnaces, thus improving the quality of the thermal environment. Existing technology for isothermal liners limits the application to 1100° C, and extension to a higher temperature range will enable materials science experiments to

properties, such as diffusion constants of dopants and thermal diffusivity in liquid semiconductors, including silicon, at well-controlled and isothermally maintained temperatures is another important application. The isothermal furnace liner is intended for installation in a module of the Materials Science Research Facility on the proposed International Space Station, but it also has immediate application to ground-based studies, where tight control of temperature will be beneficial.

The second part of this ATD project is to breadboard the equipment and determine the protocol for incorporating the hardware into future missions. In a human-tended environment, the presence of reactive liquid metals at high temperatures will demand the use of adequate containment procedures and strict protocol for the handling of the high-temperature components to ensure the safety of crewmembers.

## METHOD

A major concern in developing the high-temperature heat pipe is the need for selecting the materials for its successful construction. One of the first activities of the project was to review possible materials and examine their known history as conventional heat pipes. An extensive survey was made of a wide range of materials to determine the most appropriate and cost-effective approach for use in this project. Lithium was the clear choice for use as the working fluid. With a low melting point (181° C) and vapor pressure just below atmospheric pressure at typical required operating temperatures, its properties are ideal. Furthermore, there is an adequate database chronicling its past use in heat-transfer applications within this temperature range. Although it is not a routinely used material, purification and loading techniques of lithium for heat pipe applications are well-understood, and the technology is at a mature state.

After considerable materials evaluation, it was decided that a niobium-1% zirconium alloy would be the most suitable and cost-effective material for the body of the heat pipe. The evaluation took into account high-temperature and creep properties, reactivity, weldability, and formability. Another selection criterion was the availability of wire made

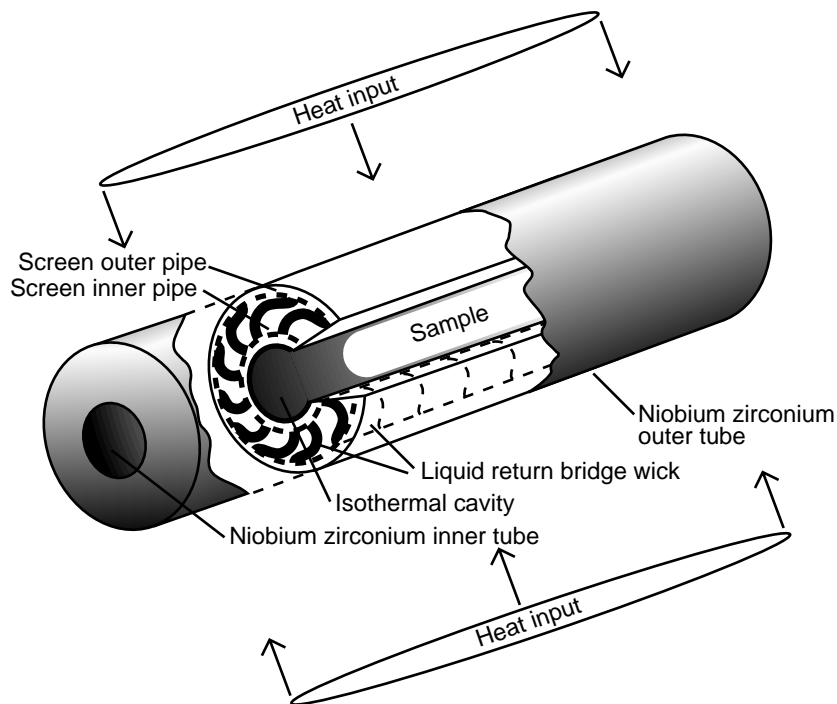


Figure 1: A cut-away schematic of the isothermal furnace liner under development

be conducted under more favorable thermal conditions. Experiments contemplated for the higher range include crystal growth by solidification of high-temperature materials, such as cadmium telluride and gallium arsenide, and application to other advanced fabrication techniques like liquid-phase sintering. The measurement of thermophysical



from this alloy for use in fabricating the screen.

A second important issue was the design of the heat pipe, both from the standpoint of function and for fabrication and operational reasons. The isothermal furnace liner has a more complex geometry than the heat transfer applications for which the heat pipe is conventionally used. This type of heat pipe, or isothermal liner, requires that the material being processed be maintained at a uniform temperature within a cavity. Therefore, the evaporator part of the heat pipe is on the outside section of an annular pipe, with the condenser at the inner section, and the wick structure is required to operate in two directions. The heat pipe assembly must first transfer sufficient heat to the condenser and maintain its temperature at a level equal to that of the evaporator. Secondly, it must maintain the same uniform temperature along the inner surface of the inner section of the pipe. The desired heat transfer is in two dimensions, with a more complex circuit for the return of the condensed liquid to the evaporator. Therefore, the wick structure has to bridge the gap from the outer tube to the wall of the inner tube, thus enabling the liquid lithium to return to the condenser. A schematic of the heat pipe construction is shown in Figure 1.

From a functional standpoint, facilitating the precise alignment of the load within the heat pipe necessitated an early design criterion specifying that this annular pipe be open at both ends, allowing one to view the positioning of the load both upon insertion and during any translation. Such a geometry puts considerably more strain on the welds that join the end caps to the main body of the heat pipe assembly. The anticipated temperature difference between the inner and outer surfaces of the pipe during heat-up will be high, particularly before the lithium vapor pressure is actively isothermalizing the assembly; thus, the potential for the welds to crack is also high. Three niobium-zirconium heat pipes have been fabricated to this design. The goal is to produce heat pipes capable of operating over long periods of time (several days) and able to withstand many cycles between room temperature and operating temperature. Testing of these heat pipes will culminate in the installation of the heat pipe in a furnace for high-temperature thermophysical property measurement and crystal growth.

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## SCHEDULE & MILESTONES

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This project, originating in fiscal year 1995 and planned as a four-year effort, originally included the development of a moving-gradient heat pipe furnace. After the first year, however, the emphasis was changed, and the effort was exclusively devoted to the fabrication and testing of the high-temperature isothermal liner. To complement the development program, dialog has been initiated to establish the protocol for incorporation of the liner as an integral part of a furnace module for the Materials Science Research Facility. The remainder of the program is devoted to extensive testing of the temperature uniformity of the inner surface of the heat pipe, the longevity of the heat pipe, and analysis of potential failure modes.

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## APPLICATION & TECHNOLOGY TRANSFER

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The instrument presented here will have a considerable impact on materials science. While specifically aimed at supervised flight applications, benefits to ground-based research are equally substantial. A high-temperature heat pipe of the specifications described does not currently exist for crystal growth, and its development will immediately produce dividends for those growing crystals and conducting other materials science research in the 1100–1500° C temperature range. In flight experiments, where investment per mission is so high, it is essential that thermal conditions for experiments be optimized so that science returns can be maximized. It is also imperative that the apparatus be robust, operational, efficient, and safe. The development of this particular heat pipe is a major step toward reaching this goal.

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# Space Bioreactor Bioproduct Recovery System

## OBJECTIVES

A major focus of NASA's microgravity biotechnology program has been the development of space bioreactors that can be used aboard spacecraft to overcome gravity-induced limitations in cell culture and tissue engineering. The current generation of space bioreactors can support some aspects of long-duration cell cultures but cannot be used to separate and preserve or remove the by-products of these processes. Some of the bioactive molecules present in trace quantities in the bioreactors are valuable, while other biomolecules present can act as cell inhibitors and either lead to termination of cellular production of desired molecules or to cell death.

The purpose of this effort is to develop a Bioproduct Recovery System (BRS) that allows the selective removal of molecules of interest from space bioreactors, thus enhancing the productivity of those bioreactors. Specifically, the BRS is designed to target specific biomolecules or waste products; to continuously adsorb and separate biomolecules from dilute bioreactor effluents; and to stabilize and preserve targeted bioproducts. The BRS will also be miniaturized, to meet volume and power constraints, and designed to operate in microgravity.

## METHOD

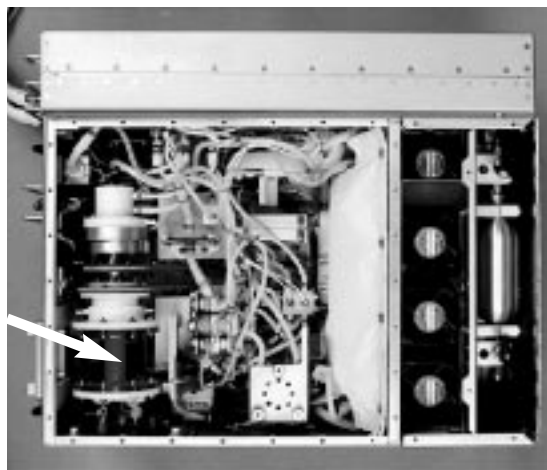


Figure 1: Top view of the NASA space bioreactor. An arrow points to the 125-ml culture vessel.

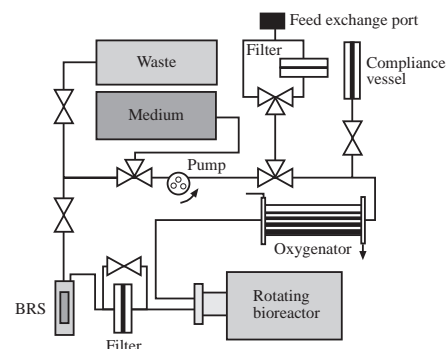


Figure 2: A schematic of the NASA space bioreactor system showing the major components

The BRS is designed for integration into the space bioreactor perfusion loop, as shown in Figure 2. Culture media from the space bioreactor flows through the perfusion loop into the BRS cartridges, each of which is packed with an adsorbent that binds, separates, and retains substantial quantities of the bioproduct(s) of interest. Each BRS contains several adsorption cartridges that contain specific-affinity adsorbents for targeted biomolecules or waste products. During operation of the bioreactor, as the BRS cartridges become fully or partially saturated with target bioproducts, they can be removed for storage or processed further. Further processing may involve flushing the saturated cartridges with solutions that either remove degradative molecules or preserve and stabilize the bound bioproducts' integrity and activity.

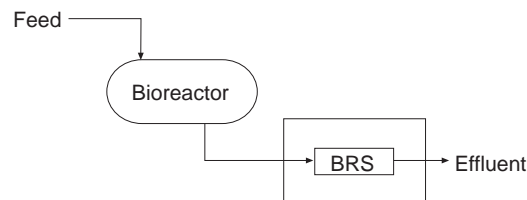


Figure 3: Downstream separation system

The BRS concept revolves around two distinct systems: an on-line system, in which bioreactor media continuously flows through the BRS cartridges, and a downstream system, in which bioreactor-spent media flows in a single pass through the BRS cartridges (Figure 3).

Each BRS cartridge (Figure 4) is packed with a solid-phase affinity adsorbent that specifically binds the target bioproduct. The capture efficiency of the BRS cartridge for the recombinant protein beta-

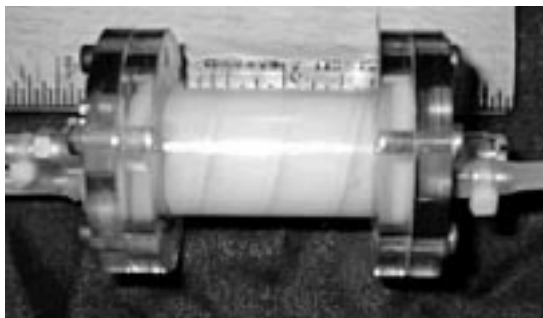


Figure 4: BRS cartridge

galactosidase for a range of perfusion rates is shown in Table 1. Compared to conventional systems, the BRS has a superior capture efficiency, especially at the higher perfusion flow rates. These high perfusion rates are often required to support the high metabolic activity and high cell densities obtained in tissue engineering experiments in the space bioreactor.

BRS Capturing Efficiency (%)		
Flow Rate (ml/min)	Conventional Column	BRS Cartridge
1	89	72
3	64	69
5	66	73
10	74	74
20	44	67

Table 1: The capture efficiency of the BRS cartridge for beta-galactosidase for a range of perfusion rates

## SCHEDULE & MILESTONES

This ATD project is funded through fiscal year 1998. One of the scheduled activities for this year is to complete the on-line evaluation in the bioreactor test bed for the three BRS cartridges that were designed and fabricated in 1997. The evaluation will be based on the capture efficiency of the adsorption cartridges developed for the first bioproduct model, the recombinant protein beta-galactosidase produced by SF-9 insect cells. The second activity is to conduct evaluations of the cartridges in parallel and in series in the bioreactor test bed. Finally, the development and evaluation of adsorption cartridges for taxol, a valuable anti-cancer bioproduct, will be initiated. The bioreactor test bed will be configured to culture conifer plant cells to produce the taxol, which will be used for the second bioproduct model.

Cell culture, bioproduct production, and bioproduct capture experiments will be conducted with these two bioproduct models and solid-phase affinity cartridge systems.

## APPLICATION & TECHNOLOGY TRANSFER

The need for separation and preservation technologies that allow the recovery of high-value biomolecules from dilute aqueous sources, such as bioreactors, has been identified by the National Research Council. In addition, the worldwide market for biotechnology-derived products is projected to exceed \$50 billion per year in the 21st century.

The use of the Bioproduct Recovery System on orbit will enhance the science returns and the commercial potential of long-duration experiments in space bioreactors in the Biotechnology Facility on the proposed International Space Station. The BRS will also be the basis of an enabling technology for NASA's Human Exploration and Development of Space Enterprise for the on-orbit recovery of valuable products from aqueous resources.

The processes and technologies being developed will drive new operational concepts, new materials and packaging, miniaturization, and new means of ensuring stability before and after use. Many of the technologies and processes developed will be used by scientists funded by the Microgravity Research Program as enabling technologies in the areas of basic biotechnology research products, disease diagnosis and therapy, and tissue engineering and creation of replacement tissues. The return of targeted biomolecules produced from long-term cultures aboard the space station will allow such molecules to be used for research, diagnostic, and therapeutics applications in the medical, health-care, and biotechnology industries.

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# Advanced Diagnostics for Combustion

## OBJECTIVES

Further understanding of microgravity combustion science demands the development of new techniques in measurement and analysis. Parameters ranging from point-temperature measurements to products of combustion are of scientific interest. Earlier techniques generally have been either intrusive to the phenomena of interest or of an insufficient quantitative nature. Recent efforts have produced new nonintrusive techniques, but a sustained increase in science quality still depends upon further improvements in measurement technology.

The goal of this project is to develop a series of more sophisticated measurement techniques applicable to the general area of microgravity combustion science. These techniques are intended to improve the accuracy and spatial/temporal yield of the data acquired and to extend the range of applicability and access to the relevant parameters presently inaccessible through current methods.



Figure 1: System components utilized for advanced laser Doppler velocimetry development

## METHOD

The content of the project is segmented into two principal areas of development: species identification and quantification, and flow-field diagnostics, addressing the characterization of complex and/or turbulent flow regimes.

### Species Identification and Quantification

- **Acousto-optic tunable filters (AOTFs)** — The objective is to provide a rapidly tunable, spectral filter for simultaneous spatially and spectrally resolved imaging applications. The AOTF combines the spectral selectivity provided by a scanning monochromator with the spatial characteristics provided by an optical bandpass filter.
- **Fourier transform infrared (FTIR) spectroscopy** — The objective is to enable the simultaneous measurement of multiple species in the

infrared. A variety of fuels and combustion products will be measured. Spectra will be acquired in both absorption and emission, accessing both ground and excited states of selected species. Band-shape analysis will provide gas-phase temperature.

- **Frequency-modulated (FM) Raman spectroscopy** — While similar to the FTIR objectives, FM-Raman permits the measurement of species such as  $O_2$ ,  $N_2$  (for temperature measurements), and  $H_2$ , which are not infrared-active. The two techniques differ also in region sampled. The FTIR is a line-of-sight method, with the additional capability of imaging; the Raman technique is a point measurement. The application of frequency-modulation techniques is intended to overcome the two traditional drawbacks to Raman spectroscopy, which are generally weak signals and the extraction of a signal against a luminous background.
- **Fiber-optic chemi-optic sensors** — The principal objective in the development of fiber-optic chemi-optic sensors is to provide a sensor array that is readily configured for the measurement of different species under difficult and varying geometric constraints. Species of interest to combustion science investigations to which these sensors could be applied include hydrocarbon fuels, oxidizers, and combustion by-products. Ultimately, the effort should result in an instrumentation package suitable for combustion experiments identified for the proposed International Space Station. As fundamentally passive sensors, they require few or no supporting electrical systems other than photodetectors.
- **Spectral data analysis software** — The primary focus of the proposed data analysis development effort is to provide software tools for determining species concentrations and temperatures from narrow-band infrared images of microgravity combustion phenomena. This effort will enhance the value of image data by using spatial correlations in the data and a general nonlinear optimizer to improve accuracy and computational speed in order to extract the maximum amount of information from the images.

### Flow-Field Diagnostics

- **Directional multicomponent laser Doppler velocimetry** — The objective of this effort is to develop and demonstrate a compact, self-contained system to perform multicomponent, directional, quantitative flow-field measurements via laser Doppler velocimetry. This effort will build on the prior development of single-component systems utilizing solid-state laser and detector technologies and dedicated high-speed digital signal processing based on LSI (large-scale integrated circuit) architectures.



- **Phosphor-based temperature-sensitive seed particles** — The proposed technology seeks to characterize and utilize temperature-sensitive phosphor materials for combustion applications. In the context of this proposal, these materials will take the form of granular particles compatible with velocimetry applications, specifically laser Doppler velocimetry.
- **Full-field turbulence quantification** — The objectives of the proposed effort include the demonstration of Hartmann-Shack wavefront sensing, as applied to turbulent flow fields, and the development of a computer code capable of measuring the wavefront slope across the field of view. This will provide the ability to quantify turbulent flow field structures as well as cell propagation velocities.

## SCHEDULE & MILESTONES

This ATD project began in fiscal year (FY) 1997. Following are the planned milestones for the currently funded areas of study:

- **Acousto-optic tunable filters:** Characterization and calibration of the spectral properties and imaging performance of AOTFs will be completed in FY 1998. The application to laboratory flames using both direct and Hadamard sampling will proceed in FY 1999. This will be followed by application and demonstration to selected reduced-gravity combustion phenomena of interest.
- **Fourier transform infrared spectroscopy:** The evaluation and selection of a spectrometer will be completed in FY 1998. Acquisition and analysis of emission and absorption data will also be initiated. Integration to support reduced-gravity applications and the upgrade to two-dimensional imaging capability will proceed in FY 1999.
- **Frequency-modulated Raman spectroscopy:** The demonstration of modulation/demodulation using CW laser sources will be conducted during FY 1998. Initial Raman measurements in laboratory combustion systems will be initiated in FY 1999, followed by application to selected reduced-gravity configurations.
- **Fiber-optic chemi-optic sensors:** The characterization and demonstration of suitable chemically reactive compounds will proceed throughout FY 1998. Emerging candidates will be integrated into fiber-optic probe assemblies. Initial application to combustion systems will commence during FY 1999.
- **Spectral data analysis software:** The initial version of analysis code will be completed and demonstrated using existing infrared emission data in FY 1998. Application to selected reduced-gravity

data will proceed during the following year.

- **Directional multicomponent laser Doppler velocimetry:** Optical and electrical characterization of newly acquired solid-state laser sources, detectors, and binary optical elements will be completed in FY 1998. Design and construction of a color-separated two-component system will be initiated in FY 1999, as will the design of a grating-based frequency shifter to provide directional capabilities.
- **Phosphor-based temperature-sensitive seed particles:** Preliminary excitation and detection strategies to facilitate testing of identified compounds under elevated temperatures and oxidizing/reducing environments will be conducted during FY 1998. Application to combustion systems and simultaneous velocity determination will be initiated during FY 1999.
- **Full-field turbulence quantification:** During FY 1998, the Hartmann-Shack instrument will be employed to study noncombusting laboratory flows with mild turbulence, such as the flow field downstream from a heated wire grid. This will be followed by application to a nonreacting heated nozzle flow from a gas jet. Application to reacting systems will be initiated in FY 1999.

## APPLICATION & TECHNOLOGY TRANSFER

In general, all of the techniques will use simple laboratory and low-gravity combustion experiments for developmental testing. While the majority of the measurement requirements are common throughout the microgravity combustion discipline, applications that will directly benefit from these developments include low-gravity aircraft investigations of solid-surface combustion and premixed-gas combustion experiments, and drop tower studies of gas-jet diffusion flames, droplet combustion, and liquid-pool fires.

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# Manufacturing of Refractory Containment Cartridges

## OBJECTIVES

This ATD project focuses on using plasma spray in a low-pressure, inert environment to form containment cartridges to be used for growing crystals of metals, alloys, and semiconductors in microgravity. This process uses high-energy (100 kW) plasma to apply layers of refractory metals and ceramics onto a graphite mandrel. A variety of materials are being characterized and evaluated against a demanding set of requirements, including high service temperature (1200–1600° C), oxidation resistance, and resistance to molten semiconductor attack. The advantage of plasma spray forming is the ability to utilize combinations of materials, such as tungsten and alumina, that are difficult to form using conventional techniques. The goal of this work is to build a database of the compatibility of 14 crystal-forming materials used by principal investigators (PIs) in the Microgravity Research Program versus 5 types of cartridge materials. This database will assist PIs in the selection of safe, reliable, and functional containment cartridges.

## METHOD

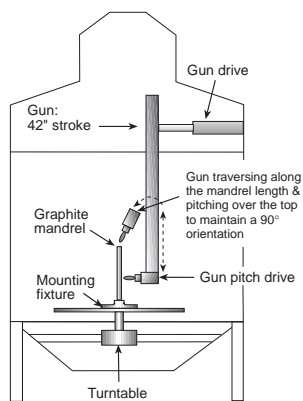


Figure 1: Vacuum Plasma Spray chamber

The fabrication sequence used to form these containment cartridges incorporates up to three layers of material in a monolithic structure. Each of these layers is deposited on a removable mandrel using the vacuum plasma spray (VPS) process. The first layer sprayed forms the inside surface of the cartridge. Its function is to contain the liquid crystal growth material should the ampoule rupture. Ceramics such as alumina and boron nitride are inert to many of the semiconductor materials used in these experiments. However, although they provide excellent chemical and oxidation resistance, ceramics are too brittle to have sufficient handling strength in the thickness

range of interest (0.65 mm). Consequently, a second layer of refractory metal is sprayed to provide bulk structural strength to the cartridge for room-temperature handling and high-temperature operation. The third layer is an oxidation-resistant material (usually ceramic) used to protect the underlying refractory metal from high-temperature oxidation.

Graphite is used for the mandrel material because of its stability at the high spray temperature and its resistance to the effects of high thermal gradients imposed by the plasma. The graphite does not metallurgically bond to the sprayed coating, allowing the coating to be pulled off the mandrel in one piece after spraying. High thermal expansion [8.6 microns/(m)(° C)] graphite is used to further ease removal, as it contracts at a greater rate than the deposit as the system cools.

During cartridge formation, a graphite mandrel is rotated about its vertical axis on a turntable while the plasma torch traverses up and down. A constant thickness of material, nominally 0.025 inch (0.65 mm), is applied over the length of the mandrel. The plasma torch is pitched from a horizontal spray axis to nearly vertical in order to coat the closed, hemispherical end of the mandrel. The photograph in Figure 2 shows the plasma torch inside the VPS chamber as it applies tungsten to a graphite mandrel. A thick buildup of material is formed at the other (bottom) end of the mandrel. This allows machining of an interface and flange at the end of the cartridge. The advantage of this forming technology over conventional processes is the elimination of two joining steps used to attach the flange to one end of the cartridge and a hemispherical cap to the other. By spray forming the cartridge as a single part, tolerances of parallelism are more easily maintained.

## SCHEDULE & MILESTONES

With the introduction of the proposed International Space Station, more opportunities will be available for materials science experiments in microgravity. This will mean more experimenters and a wider range of materials to be processed than in the past. As current cartridge materials are reaching limits to their chemical inertness and process temperatures, new and improved materials are essential for further development in the field of materials science. In general, the refractory metals and their alloys offer the chemical inertness and high melting temperatures desired for containing



materials science experiments. However, the difficulty of forming these materials into desired shapes has limited their application as cartridge materials in the past.

Parameter development and deposit characterization have been completed for a variety of refractory materials, including tungsten, molybdenum, tantalum, niobium, rhenium, alumina, and aluminum nitride/zircon. This work has been expanded to document the chemical reactivity/compatibility of 14 semiconductor or other crystalline materials with 5 refractory cartridge materials. The five refractory metal alloys used for this investigation included tungsten-25% rhenium, tungsten-3.5% nickel-1.0% iron, molybdenum-41% rhenium, tantalum-10% tungsten, and niobium-1.0% zirconium. Each of the refractory metal alloys is being heated 24 hours with each of the semiconductor/crystalline materials at the crystal growing temperature for each specific material in specially designed inert retorts at the University of Alabama, Birmingham. Energy-dispersive spectroscopy used in conjunction with the Scanning Electron Microscope showed that Mo41Re was not attacked by CdZnTe (1175° C), nor by PbSnTe (1150° C), but showed minor interaction with PbSn (600° C), major reaction with AlIn (1100° C), and severe reaction with Al/ZrO<sub>2</sub> (1140° C) and AlCu (1100° C). Testing in progress includes ZnSe, GaSb, GeSi, HgZnTe, HgCdTe, Si, and GaAs with Mo41Re. This work will be repeated with the four remaining refractory metal alloys.

## APPLICATION & TECHNOLOGY TRANSFER

The materials used by 14 PIs funded by the Microgravity Research Program have been selected to build a database of the reactivity of these materials, at their respective crystal-forming temperatures, with 5 VPS-formed refractory metal alloys. The database will not only be used to assist the current PIs in the selection of safe, reliable, and functional cartridges/crucibles for their experiments but will also be used by future PIs, from NASA and from other nations. Several PIs have expressed interest in using ceramics such as aluminum nitride (AlN) to contain their crystal growing experiments and to terminate the crystal growth by rapid quench. Ceramics, which are brittle, pose shattering hazards in handling and crack when subjected to rapid quench. However, VPS processes have

already been developed to either form refractory metal alloy cartridges/crucibles with ceramic liners using VPS or form protective metal support jackets around ceramic crucibles using VPS. Although tungsten alloys and Mo41Re alloys, unlike most metals, have coefficients of thermal expansion approaching those of ceramics, VPS techniques have been developed to transition from ceramics to metals by functional gradients, eliminating bond lines and thereby strengthening the bond between two dissimilar materials.

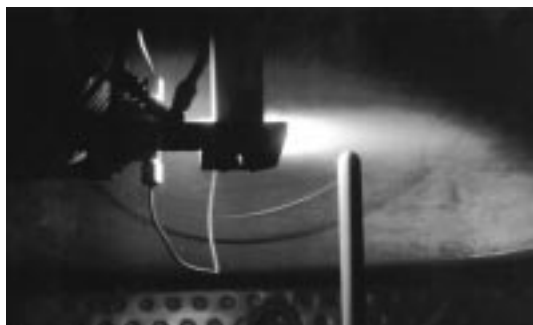


Figure 2: Photo of the inside of the VPS chamber

Taking advantage of the functional gradient transition technique developed for use in space furnaces, a project was initiated to use VPS in manufacturing a robust, low-cost, low-maintenance combustion chamber liner for the advanced X-33 and RLV rocket engines. In addition to using a new, improved VPS copper alloy, CuCrNb, a protective NiCrAlY coating will be added using the functional gradient technique. The NiCrAlY coating is calculated to reduce the operating temperature of the CuCrNb liner from 1000° F down to 800° F, or 20%, which is significant in increasing engine life and reducing maintenance costs. CuCrNb shells have been VPS-formed, cut up, and machined into test specimens that have produced very satisfactory test results to date. A small spoolpiece with a VPS-formed CuCrNb liner, representative of a rocket engine, is scheduled for hot fire testing in February 1998 to demonstrate the advanced VPS technology.

## RESEARCHER & DEVELOPMENT CENTER

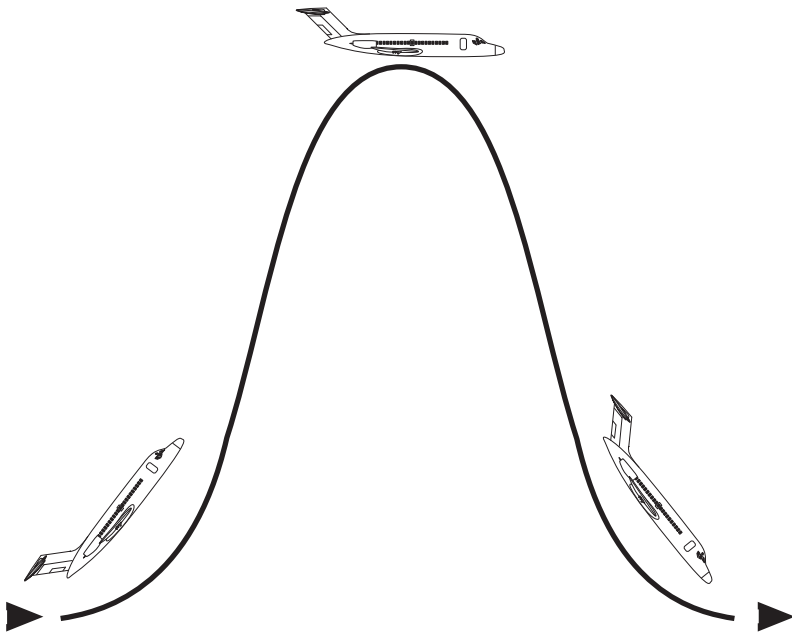
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# Free-Float Trajectory Management

## OBJECTIVES

Many researchers perform experiments that are sensitive to the effects of gravity aboard aircraft flying low-gravity trajectories. The aircraft fly in ballistic trajectories in order to attain periods of freefall, which provide the desired low-gravity (low-g) environment. These trajectories are characterized by an initial trajectory entry phase, which can be very dynamic, followed by a stabilized low-g period and, finally, an exit phase. Ideally, the entry phase should be minimized to allow the longest possible low-g time.

The low-g environment experienced by an experiment attached to the aircraft is degraded by vibrations from the aircraft, as well as by acoustic



*Figure 1: Flight trajectory of the DC-9 reduced-gravity aircraft. As the aircraft “pushes over” the top of the parabola, it experiences short periods (20–25 seconds) of freefall, or microgravity conditions.*

vibrations, disturbances caused by airflow, or self-induced disturbances. However, an experiment package allowed to free-float during the stabilized low-g phase will only be affected by direct disturbances and will not be affected by vibrations from the aircraft. The drawback of free-floating an experiment is that the package typically contacts the walls of the aircraft after only a few seconds due to the initial velocity of the package at release and the rotation of the aircraft during the parabolic

trajectory. Longer free-float times are achievable but are not predictably reproducible.

The objective of this work is to develop the technology for an extended, consistently reproducible acceleration environment during the stabilized low-g phase of the trajectory, specifically for free-float packages. The goals are to extend the free-float time to 10 seconds or longer and to obtain stable accelerations of 0.001 g or lower in a consistent, reproducible manner.

## METHOD

Improving the low-g environment for free-float packages requires the optimal control of the aircraft trajectory and the release of the package. The control of the trajectory is dependent on the limitations of the specific aircraft used and the feedback the pilots use to maneuver through the trajectory. Defining the trajectory in terms of the air speed and pitch angle upon entry, the acceleration level during pull-up, and the air speed and pitch angle upon exit of the trajectory is the first step in maximizing the overall trajectory time and, in particular, the stable low-g time.

To control the aircraft maneuver during the low-g phase, specific commands for pitch, roll, and throttle must be provided to the pilots. This requires the development of a control law to develop the appropriate commands. The control law is based on the states of the aircraft and the position of the experiment package in the aircraft cabin.

The controlled release of the experiment package once the stable low-g environment has been established will provide a consistent means of initializing the free-float period. By optimizing the initial release velocity, the package will rise off the aircraft floor and the controller will monitor its location in the aircraft. The control law will develop the appropriate control commands for the pilots to essentially fly the aircraft about the trajectory of the free-floating package. The control commands will be displayed to the pilots graphically.

The test bed for the developed technology was the DC-9 aircraft at NASA Lewis Research Center (LeRC).

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## SCHEDULE & MILESTONES

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This ATD project was initiated in fiscal year (FY) 1995 and was completed in FY 1997. The DC-9 aircraft instrumentation installations were completed and tested in December 1996. The instrumentation provides data necessary for the development of the aircraft models and for the operation of the real-time, low-gravity trajectory guidance control laws.

Aircraft parameter identification flight testing, during which the DC-9 flew standard parabolic flight maneuvers, was conducted in January 1997. The various pilot control inputs were excited in a stepwise manner, first positive, then negative, during the trajectory. The data from these flights were used to generate models of the aircraft performing the low-gravity maneuvers. The models were subsequently verified on the DC-9.

The developed aircraft models were used in conjunction with pilot dynamic models (determined previously) to design a trajectory guidance display system. Standard closed-loop control synthesis methodologies were used to determine control laws for driving the display. An aircraft flight simulation program was also designed to test and verify the controllers on the ground. The entire simulation can be operated in the aircraft using the DC-9's control stick to simulate flying a low-gravity maneuver.

The display used to direct the pilots' input consists of graphical symbols indicating the flight control positions and the desired or commanded positions. The guidance display was tested in-flight during February and March 1997. The guidance system was shown to be able to direct both the pull-up and low-gravity phases of the trajectory.

The integrated system, including position tracking and a free-floating rack, was tested for three flight weeks from April to July. Based on the results of testing, it was shown that with sufficient practice, the pilots were able to guide the aircraft during free-float and marginally improve free-float times; however, the performance was not as consistent as desired. It was concluded that in order to achieve the desired goals consistently, the guidance control system must fly the aircraft directly without pilot input. Of course, the pilot is still required as a fail safe in the event of a hardware failure. This "microgravity autopilot" could potentially fly the

aircraft through the entire trajectory, thus providing optimal initial conditions for the release of the free-floating package as well as providing consistently longer free-float times.

Unfortunately, the NASA LeRC DC-9 low-gravity research facility discontinued operations near the end of July 1997, and the potential of this autopilot was not explored. However, potential does exist to develop such a system on other low-gravity research aircraft. This ATD project did demonstrate the technology required to implement such a system.

## APPLICATION & TECHNOLOGY TRANSFER

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Numerous researchers utilize low-g aircraft trajectories to perform scientific investigations in the fields of combustion, fluid physics, and materials processing. For some, an extended high-quality, low-g free-float environment, which is the goal of this ATD project, would be sufficient for most or all of their testing needs and would reduce or eliminate the need for more expensive, time-consuming suborbital or orbital carriers. Although the developed system will only be applicable to a specific aircraft — in this case, the NASA LeRC DC-9 — the technology to develop the system will be applicable to other aircraft.

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# Passive Free-Vortex Separator

## OBJECTIVES

Effective low-power two-phase separation systems are vital for the cost-effective study and utilization of two-phase flow systems and the investigation of the governing physics of two-phase fluid flows. The fluid physics community has shown a keen interest in gas-liquid flows in microgravity for a number of years, since the study of microgravity flows has the potential to reveal significant insight into the controlling mechanisms for the behavior of flows in both normal-gravity and reduced-gravity environments. Two-phase fluid mixtures in microgravity generally require a mechanical separation device to mimic a process that occurs naturally in terrestrial environments. Commercial separator technology is not directly applicable to the requirements of spaceflight hardware because of weight, power consumption, vibration, design complexity, reliability, and volume constraints. Terrestrial passive separator technology is not valid since such devices utilize gravity to accumulate the phases after separation.

Long-duration experiments aboard either the space shuttle or the proposed International Space Station will require that consumable fluids be recycled for continuous use. The experiments require that the component fluids (gases and liquids) be separated into single-phase states prior to reuse in the experiment hardware. Once the fluids have been successfully separated, they may then be reutilized for subsequent investigations.

The objectives of the work leading to the development of the Passive Free-Vortex Separator (FVS) follow:

- To design, construct, and test a vortex separator with the capability of continuous closed-loop operation, liquid extraction and recycling capabilities, and the ability to recycle air with humidity levels that can be reasonably handled with desiccant beds (less than 60 percent) for continuous gas recycling
- To design, construct, and test a liquid drainage system based on the vortex separator with an absence of gas bubble entrainment in the effluent bulk liquid
- To design, construct, and test a vortex separator that yields a wider range of system operations and/or decreased liquid carryover and that will

be suitable for intermittent operation aboard reduced-gravity aircraft

- To use experimental data to develop and verify analytical models that will be used as design tools

## METHOD

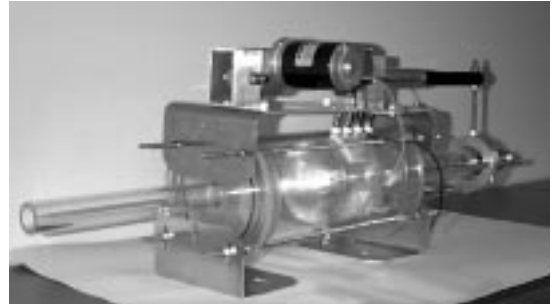


Figure 1: Mod-4 separator with actuator mechanism

The baseline FVS was developed to perform in an intermittent microgravity environment for a short duration (20 seconds) aboard reduced-gravity aircraft. This device has operated successfully during a number of operational tests with Two-Phase Flow Experiment rigs. The operation of the baseline FVS has been studied through high-speed video, and improvements have been incorporated in the system. Flight tests aboard the NASA Lewis Research Center (LeRC) DC-9 reduced-gravity aircraft have shown that the liquid carryover from the separator has been eliminated and that the system is now suitable for intermittent operation aboard reduced-gravity aircraft. The tests were conducted as a part of an ongoing scientific investigation to show that the device could operate successfully over a typical range of gas and liquid flow rates.

A ground-based test loop has been completed, and tests (including flow visualization) have been conducted to observe the structure and stability of the vortex core with a variety of different configurations. For these tests, gas is injected into the liquid flow to observe the stability of the vortex. Several designs, based on an evolutionary approach, have been tested aboard the DC-9 to validate these design concepts for microgravity operation and to examine techniques for liquid and gas extraction.



Microgravity testing has revealed the existence of four flow regimes with the separator. Core flow is when a continuous gas region forms along the axis and takes a classic vortex shape (like a tornado) that narrows as it approaches the exit. Misty core flow occurs when the vortex forms; however, there are very fine gas bubbles present in the liquid phase near the exit plate that are either migrating very slowly toward the core or trapped in recirculation zones near the exit. For bubbly flow, large spherical bubbles slowly rotate around the exit of the FVS without any defined vortex shape. For bubbly core flow, bubbles take a spherical shape but do not coalesce into a continuous gas-phase core; instead, they remain discrete bubbles as they exit the separator.

Limited analytical model development and verification to develop physical understanding of the fluid dynamics of the vortical flows and to ultimately develop design aids are proceeding. Single bubble models have been developed for both inviscid and quasiviscous flows. A test section has been designed and is in fabrication for use in validating the single and multibubble models.

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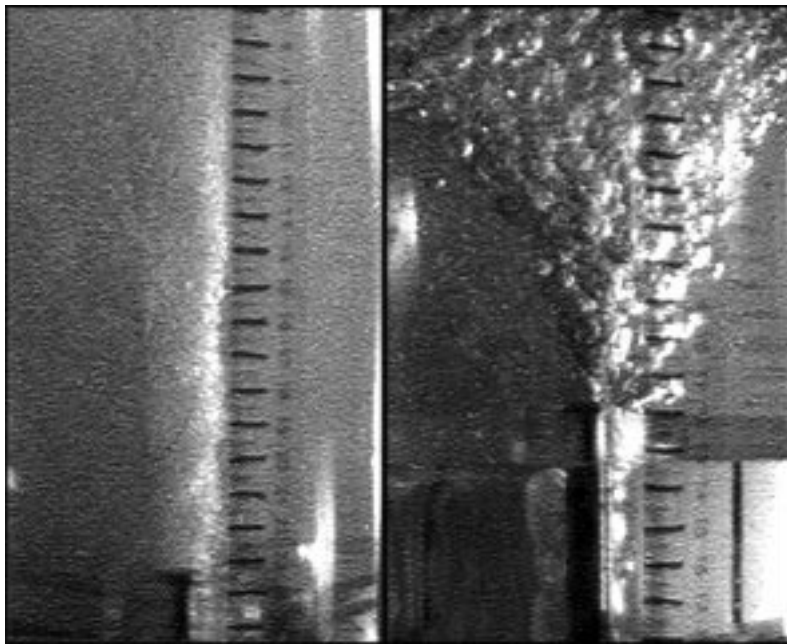
## SCHEDULE & MILESTONES

The Passive Free-Vortex Separator project began in fiscal year (FY) 1996 and was defined as a three-year effort. Development of the ground test sections was completed in FY 1996, and testing continued in FY 1997. The liquid extraction system was completed in FY 1997. The continuous recycle separator design and testing was initiated in FY 1997 and will be completed in FY 1998. Intermediate testing of designs to date will continue throughout this period as the aircraft is available.

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## APPLICATION & TECHNOLOGY TRANSFER

In addition to the traditional microgravity testing and spaceflight applications for technology transfer, potential applications for this project have been identified in the medical science community, including the separation of blood and gases. The lack of moving parts is important to this application because it reduces possible tissue damage caused by dynamic separators.



*Figure 2: Two of the four flow regimes that have been observed in the FVS during low-gravity testing. The image on the left shows core flow; the image on the right shows bubbly core flow.*

Environmental control systems, life-support systems, waste fluid handling, and fluid management in cryogenic tanks are also potential applications for the FVS device. Currently, the device is being utilized with two microgravity test rigs at LeRC, and a derivative calibrated device has been used by investigators to quantify the liquid portion of two-phase flow in experiments.

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# Laser Light Scattering With Multiple Scattering Suppression

## OBJECTIVES

Light scattering is the standard tool used to non-invasively measure diffusion coefficients of molecules or small particles in transparent fluids. In practice, measurement of the light scattering auto-correlation function is the most accurate and reliable method for determining the diffusion coefficient and, hence, the diameter of the particles in suspension. The accuracy available in a reasonable measurement time is typically about one percent or better. This technique can be applied to particle sizes extending from a few tens of angstroms (typical of small proteins) up to microns (typical of large colloids). Light scattering has become a fundamental diagnostic in two major areas of micro-gravity fluid physics experiments: colloidal systems and critical fluids. This technique has been extended so that it can now be used over a much larger range of concentrations. Diffusion coefficient and particle size information can now be extracted from solutions that range in concentration from clear to milky white.

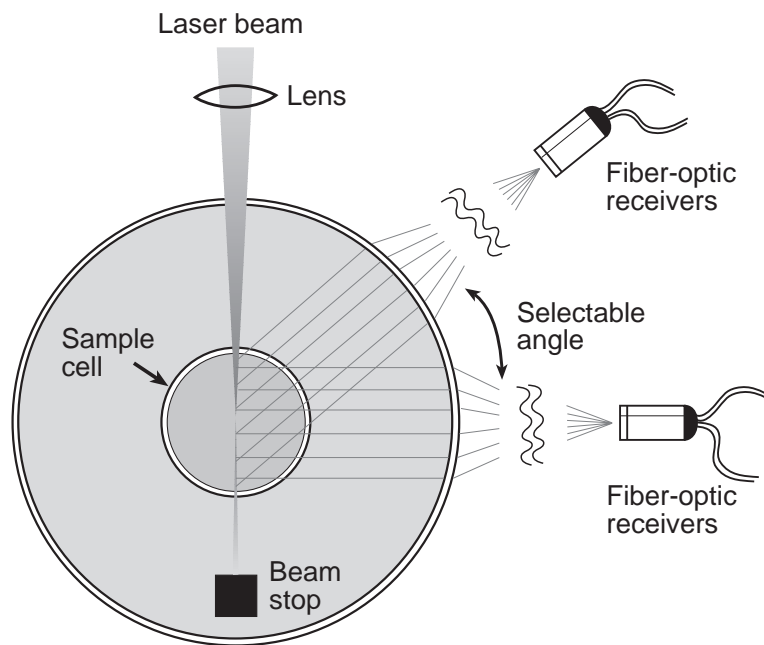


Figure 1: Optical geometry used to verify the concept of multiple scattering suppression by spatial cross-correlation

Multiple light scattering severely limits the concentration range over which laser light scattering can be used. This ATD project provides a simple and novel optical scheme that overcomes multiple scattering effects in turbid media. This ability to separate single scattering from multiple scattering has been tested in

the laboratory, and results have been published in an archival journal. Presently, the available angular range and concentration range is being increased even further by combining this technique with an extension of other developments from fiber-optic spectroscopy projects.

In addition to suppressing multiple scattering effects, ways to experimentally measure and provide a full analytical solution for double, triple, and higher-order scattering are being developed. The theoretical underpinnings of this new approach have provided an analytical solution for double scattering that includes polarization, scattering angle, and Rayleigh scattering. This solution will be generalized to  $n$ -scattering events, and Mie scattering corrections will be added to analytic solutions to the multiple scattering problem. The theory will be empirically verified, and the results will be used to interpret higher-order scattering data and to enhance the instrument design that can differentiate single scattering from higher orders of scattering.

## METHOD

The method relies on single-scattering speckle being physically larger than multiple-scattering speckle. With a suitable optical geometry (see Figure 1) to select nearby points in the far field or equivalent wave-vectors (of the same magnitude) that scatter slightly differently, the multiple scattering contribution to the cross-correlation function is suppressed. Experimental results obtained using this approach are presented in Figure 2. The basic idea hinges on the observation that single scattering originates exclusively from the region of primary illumination, which is typically the small cross section of a focused laser beam. Multiple scattering, however, arises from a broader “halo” surrounding the incident beam and thus appears to come from a significantly larger source.

Because scattered light arises from a spatially incoherent source, its spatial coherence properties are determined by the apparent dimensions of the source as viewed by the detector (van Cittert–Zernike theorem). Consequently, singly scattered light gives rise to correlated patches (time-dependent speckle) that are typically much larger in one dimension than they are in the other. They are large in the dimension in which the source appears small, namely transverse to the incident beam, and small in the dimension in which the source appears large, that is, parallel to the incident beam. Multiply scattered light also gives rise to time-dependent speckle, but since the smallest source dimension for multiple scattering is



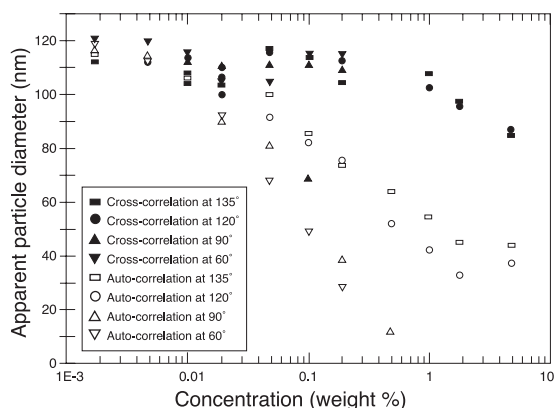


Figure 2: Particle diameter inferred from auto-correlation and cross-correlation functions using a two-cumulant fit, for measurements made at scattering angles of 60°, 90°, 120°, and 135°

larger than that for single scattering, the multiple scattering speckles are smaller than the single scattering speckles in the direction transverse to the incident beam. This approach is both simple and capable of being used at any angle. Its use of extremely low-scattering or high-scattering angles requires the addition of a promising new flare-rejection technique that is being developed and tested now.

## SCHEDULE & MILESTONES

The project's schedule for instrument development and determinations of its theoretical under-pinnings has been met and exceeded. An analytical solution for double and triple scattering has been obtained, and from it, the fiber spacing required for cross-correlation has been determined. Work to extend the analytical solution to n-scattering events is under way. Design and construction of a two-fiber cross-correlation laser light scattering instrument that uses lensless monomode fibers to detect light focused by the scattering cell at any selected angle have been completed. This instrument has been tested extensively with polystyrene standards.

The development of a new generation of high-resolution, extended delay time software correlators that will allow this technique to be used to study phenomena such as glass transitions is in progress. A very compact, power-efficient single-card correlator with analog and digital inputs for both auto- and cross-correlation has been built by an outside vendor. Work has also begun on a new type of fiber optics instrument that will address issues of flare and stray

light, which would otherwise corrupt the data. Data analysis software using Levenberg-Marquadt fits and floating baselines for interpretation of cross-correlation data has been written.

## APPLICATION & TECHNOLOGY TRANSFER

Dynamic light scattering has become a fundamental technique for gathering data in critical fluid and colloidal experiments. Critical fluid experiments test principles of universality and use dynamic light scattering to measure coherence lengths as a fluid approaches its critical point. Colloidal experiments use dynamic light scattering to measure diffusion coefficients, revealing key properties of these systems. Colloidal systems are being used to test fundamental principles of condensed matter physics and to lay groundwork for developing new materials. The advances made through this work will significantly increase the value of dynamic light scattering to the types of experiments currently using it, allow other fluids experiments to apply this technique, and facilitate the use of dynamic light scattering in materials experiments for bulk measurements for the very first time.

Currently, non-index-matched colloidal suspensions must be very dilute (essentially transparent) solutions in order for dynamic light scattering to be useful. With the advances made under this project, non-index-matched suspensions of much greater concentration can be studied. This approach will permit the use of laser light scattering to measure particle size at different depths (in fluids, eye lenses, etc.) without compromising the signal with the effects of multiple scattering when concentrations become significant. Additionally, the technique can now be used over a complete range of angles, and a method of avoiding the flare (heterodyning) issues at all angles that usually plague other methods is being tested.

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# Surface Light Scattering

## OBJECTIVES

Surface light scattering instrumentation provides a way to noninvasively measure surface tension and viscosity at the surface of a fluid interface. The fluid interface can be either liquid-liquid or liquid-vapor, and it must be optically accessible. The surface light scattering instrument described below builds upon earlier work that provided miniature, modular versions of traditional laser light and surface light scattering equipment. This project adds a new way of addressing surface sloshing that will allow the measurement of both surface tension and viscosity for transparent and optically accessible opaque media. This approach has been tested with binary fluids contained in a microKelvin temperature controller.

## METHOD

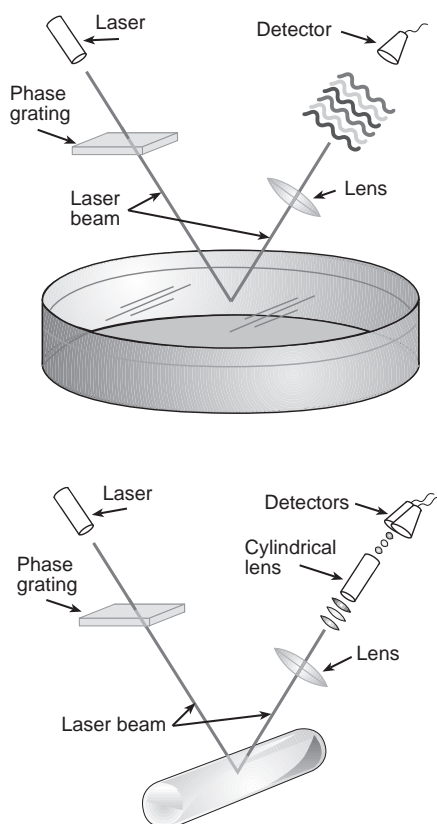


Figure 1: Surface light scattering systems. A traditional system is shown at top; below is a system using a cylindrical sample cell to reduce distortion.

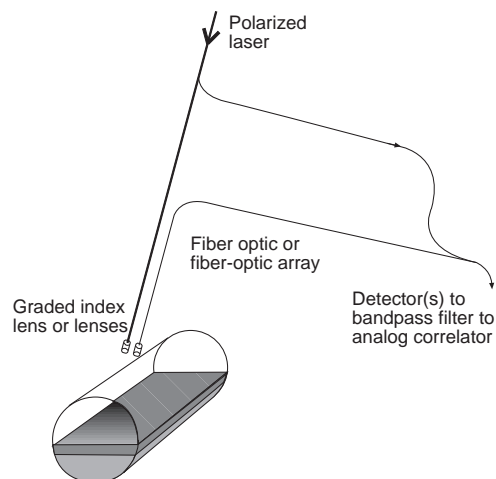


Figure 2: Reflection version of a fiber-optic surface light scattering instrument

The principle of surface light scattering (SLS) technology is the noninvasive detection of interference patterns caused by ubiquitous small surface waves called ripples, which scatter an incoming laser beam. The ripplon properties of wavelength and amplitude can be established and related to surface tension, surface viscoelastic response, and bulk viscosities — characteristics that act on the ripples as surface restoring forces and damping mechanisms. Surface temperatures may be calculated from the surface tension using established theory.

The SLS instrument systems used in this project have been developed in a building-block fashion. Several notable hardware advances have been made, including anti-slosh optical trains, analog input correlators, and a fiber optics version of the instrument. Traditional surface light scattering systems have been extremely sensitive to room vibrations. With the anti-slosh optical train shown in Figure 1, surface light scattering can be used in the presence of significant vibrations, without using vibration isolation techniques, with both transparent and opaque media. The top part of Figure 1 shows the traditional result of projecting a grating onto a fluid interface. This is done to define the scattering angle and, hence, the wavelength of the ripplon of interest. However, attempts to track the reflected first-order grating spots, which are beaten with the scattered light, are difficult at best. Reducing the diameter of the container can reduce the surface sloshing, which is the source of this problem. Yet, reducing the diameter of the fluid container introduces a distortion of the grating projected onto the fluid surface (the effect

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is much like what would be seen when viewing an image in a ball-shaped mirror). This problem can be overcome (as shown in the bottom half of Figure 1) by using a cylindrical sample cell. This allows the diameter of the container to be reduced and distorts the image of the grating along one dimension and not the other. The fact that the spacing between the grating lines remains undistorted perpendicular to the axis of the cylinder can be used to an advantage. A cylindrical lens can be used to correct for distortion in the other dimension. By using this sample container geometry, the negative effects of a sloshing interface have been mitigated, and what effects remain can be addressed by cross-correlating the first-order spots in a unique way.

Figure 2 shows a fiber optics surface light scattering instrument. Previous surface light scattering systems used bulk optics for both the introduction of the laser beam and the signal pickup. This ATD project introduced the application of fiber optics to surface light scattering instrumentation.

The new, fiber optic-based version of the surface light scattering instrument is compact, easy to align, and does not require a holographic phase grating for operation. A local oscillator is provided with a fiber coupler. The use of a GRIN (graded index) lens with a 0.5-micron core polarization-maintaining fiber makes the instrument highly sensitive to the angle of the incoming light, causing the fiber to act as a stop that rejects stray light. This instrument requires the use of anti-slosh optics to minimize instrument broadening and can be combined with our new anti-slosh cell and cross-correlation techniques.

The SLS project has also worked to develop ways to measure the power spectrum of large amplitude surface waves. The immediate application is the levitated liquid drop experiments at NASA's Jet Propulsion Laboratory. By adapting commercially available laser vibrometer technology, successful measurements on driven surface oscillations with amplitudes as high as 700 microns have been made. This will permit study of turbulent and nonlinear flow regimes on the surface of levitated droplets.

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## SCHEDULE & MILESTONES

A new kind of anti-slosh surface light scattering instrument has been designed and built to work on all types of materials. Initial tests using cells installed in a modified critical point facility show this new design to be very easy to use and able to

accommodate a wide range of materials. When the same optical train was used with fluids contained in a conventional flat dish, the data degraded considerably (low-frequency noise was present because the surface sloshed with several degrees of freedom). Last year, the instrument was tested on simple fluids. This year, in cooperation with the University of Amsterdam, successful tests have been conducted on binary fluids both above and below their critical point. The anti-sloshing techniques designed into this instrument include a new cross-correlation technique, and the instrument is proving to be quite robust.

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## APPLICATION & TECHNOLOGY TRANSFER

Surface tension is an elusive phenomenon that controls many individual processes and everyday phenomena. It is the two-dimensional analog of pressure, and it acts to maintain the smallest possible surface area. It affects cooking, cosmetics, tertiary oil recovery, detergents, controlled-release and targeted drug delivery, materials processing such as steel making, and many other activities as well. The study of surface tension-driven phenomena is often masked by gravitational forces that are not present in the reduced-gravity environment of a space station or space shuttle. This instrument provides not only a noninvasive measurement of surface tension from which surface temperature can be extracted, it also gives viscosity information. Viscosity is the internal friction of a fluid, and it affects various substances, such as the liquid crystal displays used in flat-screen computer monitors.

Space experiments that could benefit from surface light scattering include critical point studies, surface wetting experiments, free-surface phenomena experiments, pool boiling experiments, surface tension-induced instabilities experiments, and surface tension-driven convection experiments.

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# The Laser-Feedback Interferometer

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## OBJECTIVES

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The objectives of this ATD project are to evaluate, adapt, and deliver a novel form of interferometry, based upon laser-feedback techniques, that will provide a robust, versatile, state-of-the-art diagnostic instrument applicable to a wide variety of micro-gravity fluid physics and transport phenomena.

Laser-feedback interferometry (LFI) differs from conventional interferometry in that it uses the laser as both a light source and a phase detector. Either a cavity or a semiconductor (diode) laser can be used. LFI can be used either in direct reflection or by passing the interrogating beam through the sample and then reflecting it into the laser. A major benefit of LFI compared to other forms of interferometry is that high signal-to-noise ratios can be obtained at significantly lower levels of modulation of the incident laser light.

The instrument developed under this ATD project can be used to measure both temporal and spatial changes in optical path length and object reflectivity. Phenomena that vary slowly over time and dynamic phenomena can be measured over both microscopic and macroscopic fields-of-view. Since the data from the interferometer can be integrated, there is no upper limit to the range of the measured displacement. Additionally, the direction of the displacement can be determined unambiguously. The apparatus can accommodate small and large working distances (up to many cms). For a microscopic field of view, the laser-feedback interferometer has been incorporated with a reflected-light optical microscope and has also been combined with long working-distance objectives.

## METHOD

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This ATD project consists of a multiyear effort to accomplish three specific goals:

- Build a phase-shifted laser-feedback interferometer, based upon a continuous-wave HeNe laser with stationary imaging optics and a translated sample; quantify the random and systematic measurement errors (including sensitivity to external perturbations); and calibrate the technique by measuring the cantilever bending of a piezoelectric bimorph and measuring the static contact angle for a Newtonian fluid

- Modify the two-dimensional scanning technique so that the sample remains stationary and the optics translate
- Investigate the LFI response with semiconductor diode lasers and incorporate a diode laser with fiber coupling into the previously designed instrument

To ensure that the apparatus meets the science requirements of the diverse microgravity research community whenever possible, other fluid physics researchers will be encouraged to participate in the various phases of this project and to utilize the technology.

## SCHEDULE & MILESTONES

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This ATD project began in fiscal year 1995. Laser-feedback interferometry has been combined with the principles of phase-shifting interferometry to produce a novel instrument that can simultaneously measure the variation in optical path length and discern sample reflectivity variations. The accuracy and precision of our technique has been determined by measuring the cantilever bending of a piezoelectric bimorph, the phase change in an electro-optic modulator, and the contour of static drops of oil on coated substrates. In addition, the response of the instrument has been characterized for high- and low-reflectivity samples. The instrument has been incorporated in a reflected-light microscope and used with high numerical aperture objectives. The ability of the instrument to measure bending and vibrational displacements (in a noisy environment with minimal vibration isolation) was demonstrated at NASA Tech 2005 in October 1995. During the final year of the project, beam scanning over a fixed sample and the incorporation of a laser diode as the source will be completed. Also, measurements of thin films of Newtonian and non-Newtonian fluids will be conducted.

Figure 1 shows the change in the optical path length due to the bending of a piezoelectric bimorph (a sandwich of two piezoelectric elements of equal but opposite piezoelectric materials). The bimorph is clamped in a vise, forming a cantilever beam, and a voltage is applied across the elements. As shown, the bending is quadratic as a function of the distance from the clamped end and changes direction when the polarity of the DC voltage is changed. The bending is shown for two applied

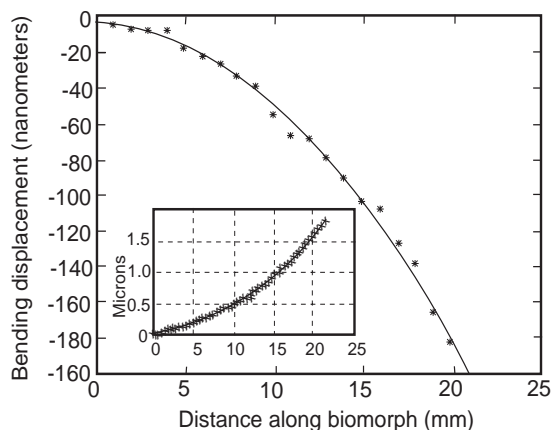


Figure 1: Bending curve for a piezoelectric biomorph for two separate voltages obtained using phase-shifted laser-feedback interferometry: 4.0 V (inset) and -500 mV

voltages, 4.0 V (inset) and -500 mV. This demonstrates that phase-shifted laser-feedback interferometry can be used to make accurate measurements of the change in optical path length.

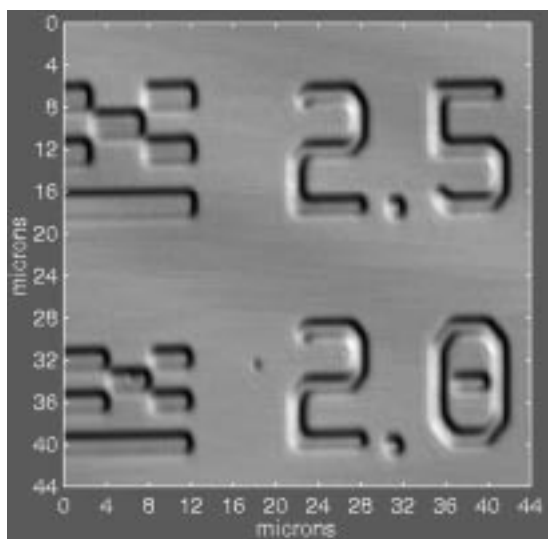


Figure 2: Variation of the reflectivity across a silicon reference target. The 75-nm-high bars have pitches of 2.5 microns and 2.0 microns.

In Figure 2, an image of a silicon reference target obtained with the phase-shifted laser-feedback microscope is shown. The numbers indicate that the bars (approximately 75 nm high) have pitches of 2.5 microns and 2.0 microns.

## APPLICATION & TECHNOLOGY TRANSFER

Often the primary science requirements of experiments of relevance to the Microgravity Research Program, both ground-based and flight-based, involve the measurement of phenomena that can be assessed by measuring the spatial variations of the optical path length of an interrogating light ray. These variations can arise either from changes in the index of refraction along the path of the ray (a function of temperature, pressure, mass density, and concentration) or from changes in the distance a ray travels.

Some examples of science requirements from experiments of relevance to the microgravity fluid physics discipline that can be determined from accurate measurements of the changes in sample reflectivity and optical path length include determination of the location and orientation of a contact line and interface shape between two fluids; the evolution of the thickness of a thin film; deformation of a free surface due to evaporation or vibration; resonant mode shapes in bubbles; and variations in temperature, density, surface tension, viscosity, and diffusion coefficients in fluids. Measurement of the structure of growing protein crystals is another application from a microgravity research discipline.

The developed technology is likely to be attractive to many users outside the aerospace community. In fact, LFI will augment the ubiquitous uses of interferometry that have demonstrated applicability to nearly every scientific discipline, including physics, materials science, mechanical engineering, chemistry, and biomedical engineering.

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# Development of an Electrostrictive Valve

## OBJECTIVES

The objectives of this ATD project are to develop a miniature cold valve with no moving parts for use as an active phase separator for liquid helium and to study the ability of a submicron aperture to act as a tunable Josephson junction in  $^4\text{He}$ .

Liquid helium is used as a cryogen in many spaceflight experiments. The use of liquid helium in microgravity is complicated by the need to separate the gas and liquid phases, which is normally accomplished by gravity. The evaporation rate of helium is controlled by the device used as a separator. If the evaporation rate is too low, the temperature of an experiment rises; if it is too high, the lifetime of the experiment is shortened. Currently, the separation is accomplished with a porous plug between the helium tank and the vacuum of space. The main disadvantage of the porous plug is that it is unregulated and, to some extent, unpredictable. It is also rather bulky. Some effort has been put into development of active phase separators that would control the evaporation rate of the liquid helium by changing the width or length of a narrow flow channel. These phase separators are also bulky and contain moving parts, reducing their reliability. In addition, they are usually actuated by a solenoid and thus require substantial electrical current for their operation. This may lead to unacceptably large heat loads in long-lifetime experiments.

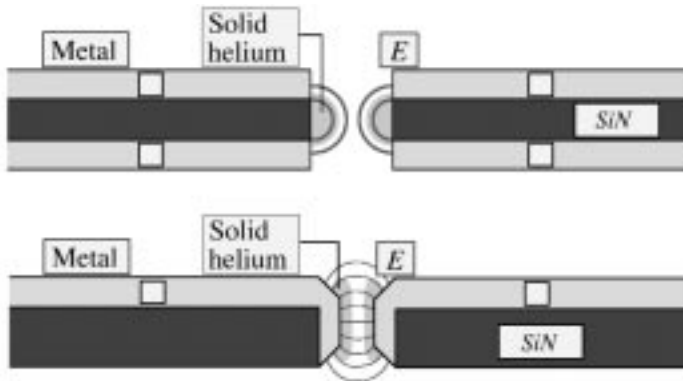


Figure 1: Schematic of the electrostrictive valve and two possible electrode configurations

## METHOD

This ATD project is focused on the development of an active phase separator based on

electrostriction in helium. When an electric field,  $E$ , is applied to part of the fluid, the pressure is increased in this part by

$$\frac{(e - 1) \cdot e_0 \cdot E^2}{2}$$

where  $e$  is the dielectric constant of the fluid (1.055 for liquid helium). Helium remains liquid at low temperatures up to a pressure of 24 bar and solidifies above this pressure. If an electric field of sufficient strength is applied to the liquid helium in a narrow gap, it is possible to solidify it, thus blocking the gap. If the field is not homogeneous, it is possible to block only part of the passage, where the field strength is higher. Thus, by controlling the electric field, we can control the impedance of the passage.

The main unanswered question is whether a field of sufficient strength can be applied without electric breakdown of the helium. Most breakdown field strength measurements in liquid helium have been performed with centimeter-sized electrodes. The results show that the maximum field one can achieve is approximately 100 MV/m, if the electrodes are immersed in the liquid. This is insufficient to solidify helium. On the other hand, measurements with needles with submicron-sized tips show that voltages as high as 2.3 GVolt/m can be achieved. This is close to the value that is required to freeze the helium (3.4 GVolt/m). The question of attainability of the required electric field will be answered by this study.

The proposed valve consists of an aperture in a silicon-nitride (SiN) membrane that is about  $0.1 \mu\text{m}$  thick. The aperture will be a slit that measures  $0.2 \mu\text{m}$  by  $10 \mu\text{m}$ . The electrodes for applying an electric field will be formed by thin films deposited either on both sides of the membrane or on one side of the membrane on two opposite banks of the slit. A high electric field will be applied to the helium in the slit whenever voltage is applied between the electrodes. This will cause the helium in the slit to solidify.

The proposed valve will be miniature, will have no moving parts, and will be able to be actuated by a moderate voltage with practically no power dissipation. It is particularly suitable for application on miniature spacecraft. The valve will be manufactured on a silicon wafer. The whole temperature-regulation scheme can be manufactured on the same wafer, resulting in an extremely compact design.

The important scientific application of this project will be design of a variable-size submicron aperture. Such an aperture can be used to demonstrate Josephson junction behavior in liquid  $^4\text{He}$  and to study size effects in the material. At low temperature, coherence length is very small ( $\sim 1 \text{ \AA}$ ); as the temperature of liquid helium approaches the lambda transition point, it increases. When the coherence length becomes comparable with the size of a flow path, Josephson junction-type behavior is expected to be observed. Pressure change caused by application of electric field in the aperture will shift the transition temperature, thus allowing the parameters of the junction to be tuned. By suppressing the transition temperature to below the temperature of the experimental cell, we can study the proximity effect. The proximity effect is the induction of superfluidity or superconductivity in otherwise normal media by nearby superfluid or superconducting regions.

This experiment will require microgravity in order to approach the lambda transition point of helium closely enough.

## SCHEDULE & MILESTONES

The project was initiated in fiscal year (FY) 1997, but the development progressed slower than expected due to lack of manpower. However, most of the FY 1997 milestones were met. Milestones reached include material procurement, formation of SiN windows, obtaining results of SiN electrical breakdown tests, and manufacturing the aperture in the SiN membrane. Milestones remaining include obtaining the results of helium breakdown tests, obtaining results of the flow tests, submitting a publication on superfluid transition temperature suppression, and completing a review of the project.

Due to the aforementioned lack of manpower, the project spent only about half of the FY 1997 budget. The work will be continued in FY 1998.

## APPLICATION & TECHNOLOGY TRANSFER

The proposed technology is beneficial in many ways. It will fill four technology niches by providing an active helium phase separator, a cryovalve, a helium superflow Josephson junction, and a superfluid gyroscope. The field of flight cryogenic engineering will benefit from the development of a new phase separator with controlled cooling power,

smaller size, and lighter weight. Ground-based and flight-based condensed matter researchers, particularly in helium physics, will be able to use the technology to their advantage, and there are currently several NRA-funded ground-based science activities that can benefit directly from the electrostrictive valve. In addition, new research could be conducted with the use of the electrostrictive valve as a Josephson junction in superfluid helium, and a superfluid gyroscope could be created as a spin-off of the development of a Josephson junction.

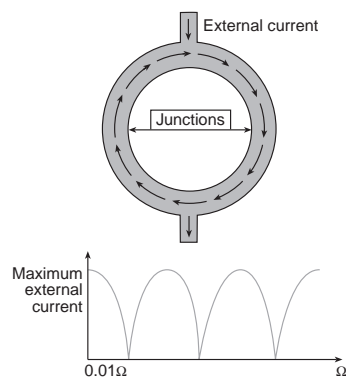


Figure 2: Schematic of the superfluid gyroscope

Two Josephson junctions connected in a loop (as is shown in Figure 2) form an analog of a superconducting quantum interference device (SQUID). Just as a SQUID is extremely sensitive to a magnetic field, the helium device will be extremely sensitive to its rotation. Rotation will modulate the maximum external current that can be passed through the device, enabling it to function as a very sensitive gyroscope. The potential sensitivity of such a gyroscope is higher than that of a laser gyroscope by the ratio of the mass of a  $^4\text{He}$  atom to the mass of a photon, a factor on the order of  $10^9$ . Ultrasensitive gyroscopes are used, for example, in geophysics for extremely precise measurements of the rotation of the Earth. The proposed device can be miniaturized, although some sensitivity will be sacrificed.

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# A Protein Crystal Growth Studies Cell

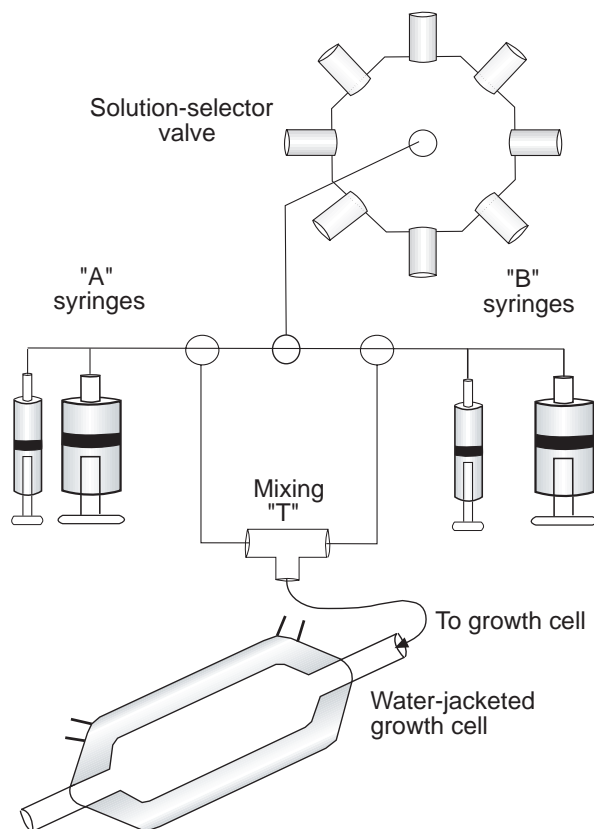
## OBJECTIVES

Current microgravity protein crystal growth (PCG) hardware systems are hybrid, attempting to serve two purposes: (1) acquire data about the processes of crystallization and (2) grow crystals suitable for X-ray diffraction studies. This duality of purpose leads to compromises in system design, with the result that neither purpose is successfully served. While structural studies of protein crystals require the best nucleation and growth conditions for each crystal growth cell, as well as the return of the crystals to Earth for analysis, the study of the growth process requires that every solution parameter be rigorously controlled and measurable for maximum data collection. Current hardware relies on a “one-shot” design, with all solution

parameters defined prior to launch and a return to Earth between experiments, which impairs the study of the growth process itself.

The primary goal of this project was to design and construct prototype cells and associated systems for the study of the PCG process. A single-cell design, suitable for studying solution concentration gradients surrounding a growing crystal, local interfacial features of protein crystals under growth and etching conditions, and simple averaged face growth-rate measurements, was developed. The system is capable of making all measurements under either quiescent or flowing solution conditions. A second goal of the proposed work was to develop practical methods for storing proteins prior to use in crystal growth and other experiments. The design of the growth cell and associated fluidic systems in this project was based upon obtaining the following target characteristics:

- Growth cells suitable for specific study goals
- Temperature control of the growth cell from 0–40° C ( $\pm 0.05^\circ$ )
- Control of nucleation and growth of crystals at a defined location, or ease of centering a specific crystal within the observation region
- A fluidic system that accurately prepares crystal growth solutions from stock solutions and delivers them to the growth cell
- Cells accessible for additions to and/or modifications of the solution
- The ability to do follow-up experiments based upon preceding growth results
- Some cell adjustment to bring selected crystal faces in line (perpendicular or parallel) with the optical axes
- Easy accessibility to other solution measurement systems (pH, conductivity, etc.) for maximum data return
- Capability of being remotely operated from the ground to the maximum extent possible
- Ability to maintain proteins in a viable state prior to use



*Figure 1: This diagram shows the system for preparing a cell for protein crystal growth. Each valve port is used to prepare one-half of the crystal growth solution. The final solution is freshly mixed just prior to injection into the growth cell. More solution components, or a larger number of proteins, can be accommodated by the addition of more eight-port solution-selector valves.*

## METHOD

Initial cell designs were found to have too many design flaws for facile use, including problems with replacing the solution within the cell without introducing bubbles, and difficulty in designing optical systems to pass through a curved glass interface. Subsequently, a two-cell approach was tried, with

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one cell optimized for holographic data collection and the other for interferometric data collection. However, as ground-based data analysis methods are most likely to employ interferometry, the limiting resolution in this case would have come from the holograms collected by the cell. Accordingly, a single-cell design approach, optimized for interferometric data collection methods, was implemented. Several growth cells were fabricated and tested, the largest having a rectangular cross section of 2.0 cm x 1.0 cm x 0.1 cm. The small fluid volume in this case reduces the amount of solution needed for a given experiment. This cell is currently being used in PCG experiments with an existing crystal growth-rate measurement system.

The target thermal regulation for the initial design was 0.1° C. A thermal regulation system using resistance heaters able to actively regulate a growth cell to within 0.015° C was devised. Operation at ambient +5° C or lower (down to 0° C) requires active cooling. This led to an investigation of the use of a circulating fluid bath to control temperature, rather than actively heating and cooling the cell. This arrangement is beneficial, as the waste heat from the cooling system can be dumped away from the growth cell and associated optics.

A fluidic system has been assembled and connected to the growth cell (see Figure 1). This system can prepare separate protein and precipitant solutions from up to eight component solutions. The protein and precipitant solutions are held separately, with mixing occurring immediately prior to injection into the growth cell. More complex solutions, or expansion of the system to handle several different PCG systems, can be easily implemented. The assembled fluidic system with growth cell has been integrated into a growth-rate system and will be used for automatic seed crystal and growth solution preparation. All operations of the growth-rate system are controlled remotely through a personal computer.

The ability to store solutions, particularly protein solutions, so that they can be used over a period of several months is of primary importance. Storage stability experiments with model proteins to determine optimal conditions were conducted. The proteins were stored as solutions and as freeze-dried powders at 20°, 4°, and -20° C and were tested biweekly for crystallizability. Storage in the frozen or freeze-dried states was found to lead to more stable proteins and

is preferred on Earth. However, difficulties in re-mixing thawed solutions and removing small amounts of cold precipitated protein will necessitate some manual intervention if frozen or freeze-dried materials are used in microgravity. Currently, storage as a solution at 4° C appears to be the most satisfactory approach, especially as the most likely model proteins to be used in near-term PCG studies are those that are both plentiful and relatively stable.

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## **SCHEDULE & MILESTONES**

This was a three-year project started in fiscal year 1995. The work is a follow-up of a previous one-year proof-of-concept effort demonstrating the close thermal regulation of a small fluid-volume PCG cell.

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## **APPLICATION & TECHNOLOGY TRANSFER**

Using this PCG cell, researchers will be able to monitor and change selected solution parameters to optimize both experimental conditions and the return of experiment data. This will enable the study of microgravity effects on the PCG process and investigations into how the process can be improved both in space and on Earth. Long-term protein solution storage and stability will become a concern as the timescale of microgravity experiments increases. Developing methods for long-term protein storage will improve both this and future microgravity-based instrumentation.

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# High-Resolution Thermometry and Improved SQUID Readout

## OBJECTIVES

One very active area of research in condensed matter physics is the study of critical phenomena and phase transitions. The success of critical point experiments depends on both measuring and controlling the thermodynamic state of the test sample with very high precision. Particularly in the microgravity environment, the precision with which temperature and/or pressure can be measured often sets the limit for science return.

The current state-of-the-art high-resolution thermometers (HRTs) use paramagnetic salts to sense temperature and superconducting quantum interference devices (SQUIDs) to read out the signal. These thermometers have relatively large heat capacity and usually cannot be in direct thermal contact with a test sample. As a result, their performance can be degraded by thermal fluctuations and, in the space environment, by particle radiation. The goal of this project is to overcome these problems by developing a high-resolution penetration depth thermometer (PDT) using a two-stage series array SQUID amplifier (TSSA) for readout. The PDT uses a thin superconducting film as the active element and can have very low mass and heat capacity. In contrast to HRTs, a PDT can be made in a variety of geometries, allowing greater flexibility in experiment design and better thermal coupling. The

TSSA uses an array of up to 200 SQUIDs as an amplifier for a single input SQUID. This arrangement simplifies the room-temperature electronics required and leads to better energy resolution by an order of magnitude. The TSSA is also designed to be relatively insensitive to external magnetic fields and electromagnetic interference (EMI). These features make it very attractive for spaceflight applications by reducing both cost and shielding requirements.

Using a TSSA for readout, the ultimate sensitivity of a PDT is  $10^{-11}$  K/ $\sqrt{\text{Hz}}$ . This figure is roughly independent of the thickness and type of superconductor used. Since a large number of microgravity experiments are being planned to probe the superfluid transition in liquid helium, the goal of this project was to demonstrate  $10^{-10}$  K/ $\sqrt{\text{Hz}}$  resolution near the lambda point of helium (2.177 K).

## METHOD

One defining property of superconductors is their ability to prevent the penetration of external magnetic fields into their interior. In a superconducting film thinner than the London penetration depth, the effect leads to only a partial attenuation of external fields. Because the attenuation varies with temperature, a coil located near a superconducting film in the PDT will exhibit a temperature-dependent inductance. The coil is connected in series with a SQUID amplifier to read out changes

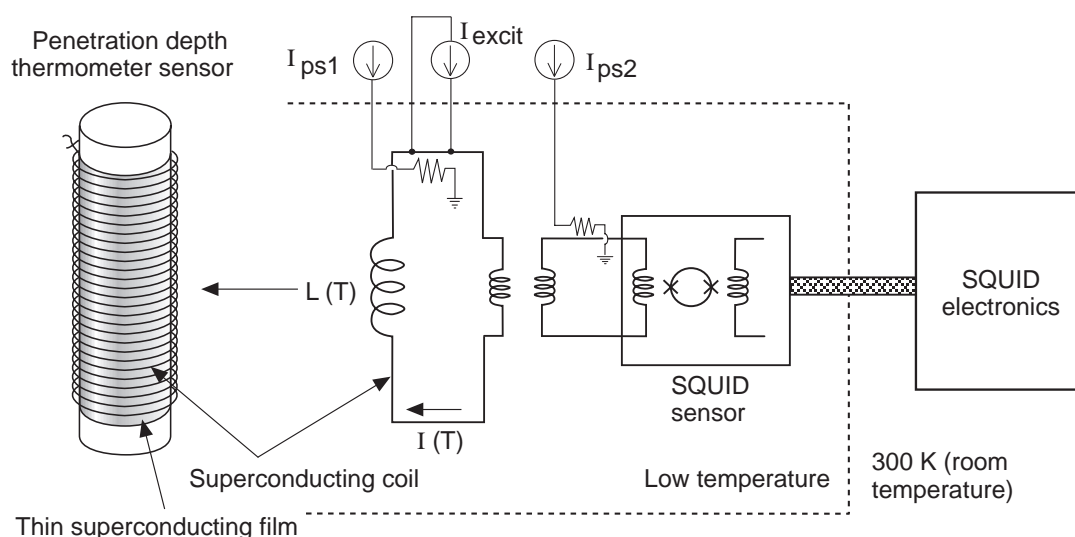


Figure 1: Circuit for excitation and readout of PDT sensors.  $I_{excit}$  and  $I_{ps1,2}$  are the excitation and persistent switch heater currents.



in inductance and, hence, temperature. Resolution is determined by a number of factors, including coil configuration, film geometry and thickness, substrate material, and SQUID current resolution. Aluminum films will be used for this work since the transition temperature can be varied by adjusting film thickness. This will facilitate developing a PDT with maximum sensitivity at 2.177 K. Other parameters will also be varied to obtain optimum performance.

Development of the TSSA will focus on eliminating extrinsic noise inputs and breadboarding feedback control electronics, which are needed to linearize the output and increase the dynamic range. These electronics may be able to incorporate 1/f noise-reduction techniques used in commercial systems. They will be tested for sensitivity to EMI. The goal is to produce a flight-qualifiable SQUID system with better energy resolution by at least a factor of two over existing commercial systems at frequencies down to 1 Hz, improving to a factor of 10 better above 500 Hz.

## SCHEDULE & MILESTONES

This project was initiated in fiscal year (FY) 1994 and ended in FY 1997. Following are the significant milestones of the project:

- Sensors that operate near the lambda point of helium (2.177 K) have been fabricated and measured to have intrinsic sensitivities of  $3 \times 10^{-10}$  K/ $\sqrt{\text{Hz}}$ .
- A thermally stable platform that allows test thermometers to be immersed in superfluid helium was built. A combination of good thermal isolation and the high heat capacity and thermal conductivity of the helium will provide an environment that is stable to better than  $10^{-11}$  K/ $\sqrt{\text{Hz}}$ .
- The best temperature resolution of a PDT is 2 nK/ $\sqrt{\text{Hz}}$  at frequencies above about 200 Hz. At lower frequency, excess noise is observed with a 1/f frequency dependence. PDTs are therefore well-suited for AC temperature measurements and could be used for such devices as a second sound detector or as a bolometer for detecting radiation. Having low heat capacity and high thermal conductivity, the response time of a PDT can be less than 1 microsecond.
- The TSSA chips developed achieve less than 2 pA/ $\sqrt{\text{Hz}}$  current resolution at frequencies above 200 Hz, with 1/f noise dominating below about

10 Hz. The TSSAs have small input inductance ( $\sim 0.15$   $\mu\text{H}$ ), and therefore have very high bandwidths, above 1 MHz.

- A direct-coupled feedback controller has been developed that allows for easy biasing and control of TSSA chips in a high dynamic range ( $>10^6/\sqrt{\text{Hz}}$ ) and moderate bandwidth ( $>10$  kHz). The electronics can easily be re-optimized to increase the bandwidth to above 1 MHz, with some degradation in low-frequency performance ( $<100$  Hz). The total system resolution is 2 pA/ $\sqrt{\text{Hz}}$ .

## APPLICATION & TECHNOLOGY TRANSFER

These technologies are being developed to aid a growing number of flight experiments in micro-gravity that use high-resolution thermometers and pressure sensors, and that use SQUIDs for sensor readout. The PDT can be used both as a thermometer and as a fast-response thermal detector, with such applications as the detection of second sound waves in superfluid helium. The TSSA can be used in any application requiring SQUIDs but is best suited to measuring signals at medium to high frequency, where its sensitivity exceeds that of commercial SQUID systems. In applications where measurement resolution is not limited by SQUID noise, the TSSA can be implemented at considerably lower cost than commercial systems due to its simplified control electronics. With an extremely high bandwidth ( $>1$  MHz), the TSSA can be adapted to multiplex large numbers of SQUID channels and could be used with such systems as magneto-encephalography or detector arrays.

TSSA chips are commercially available from HYPRES, Inc., Elmsford, NY 10523.

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# Application of Superconducting Cavities to Microgravity Research

## OBJECTIVES

This ATD project has two main objectives: (1) to use modern microwave electronics, high-Q low-temperature superconducting cavities, and high-resolution temperature control to develop an ultra-stable oscillator system that will provide a comparison oscillator for the laser-cooled atomic oscillators now under development in the Microgravity Research Program; and (2) to develop high-temperature superconductor (HTSC) materials, high-Q cavities, and electronics that can be integrated with a small cryo-cooler to provide a user-friendly materials characterization system for use on the proposed International Space Station (ISS). The ultra-stable oscillator will strive for frequency stabilities better than  $df/f = 10^{-16}$  to provide comparison signals unattainable by other means. The HTSC materials characterization system will provide high-resolution sample characterization if the HTSC cavity obtains Q values greater than  $10^6$ . Both of these superconducting cavity systems will better benefit users on the ISS if they can be cooled with small cryo-coolers that are capable of long-term operation in microgravity.

## METHOD

Conceptually, the ultrastable oscillator is very simple. A frequency synthesizer will generate a

signal near the resonant frequency of a high-Q superconducting cavity. That signal will be sent to the cavity with waveguides or coaxial cables, and the reflected signal will be compared to the synthesizer signal. If the synthesizer signal frequency is right at the cavity resonance, no reflection will be seen. When it is off-resonance, the properties of the reflection will indicate how to correct the synthesizer frequency to move toward the cavity frequency. The correction procedure will utilize modulation sidebands on the synthesizer signal and will actually be set up to act continuously in a phase-locked loop (as shown in Figure 1), so the synthesizer will be locked to the resonant signal of the high-Q cavity. Using low-noise electronics in the synthesizer and making the environment of the cavity very stable will lead to an ultrastable oscillator system.

The sample characterization system is also conceptually simple. A sample will be placed in a superconducting cavity, and the increase in absorption of the microwave signal caused by the sample will be measured. Samples with low impurity levels and a high degree of crystal perfection will yield lower absorption levels. The better the quality factor, Q, of the cavity, the more sensitive the characterization system is to sample imperfections. The system under development will employ HTSC materials for the microwave cavity so that it can be easily cooled to temperatures where the cavity attains a large Q.

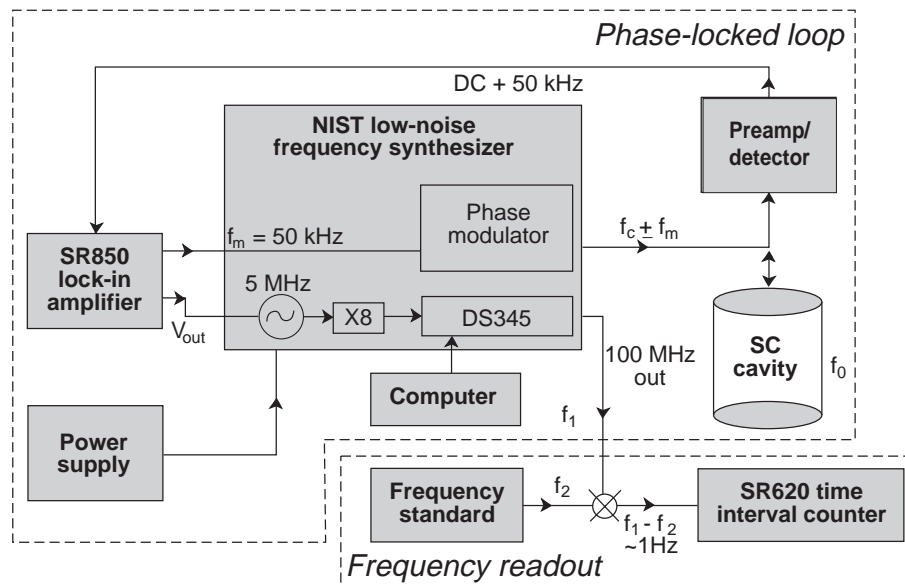


Figure 1: Block diagram of frequency measurement using the phase-locked loop technique

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## SCHEDULE & MILESTONES

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### Ultrastable Oscillator

This task is planned to produce a stable oscillator signal in the third year of its development. During the remainder of the four-year period, it will demonstrate improved stabilities until it reaches its stability goal of  $df/f$  in the  $10^{-17}$  range. During the third and fourth years, the oscillator system will be made available to developers of other oscillators to perform comparisons between stable oscillator systems.

This task began in fiscal year 1997. During the first year, design of the oscillator was performed, and a source of the low-noise synthesizer electronics was located. The synthesizer will be supplied by the Frequency and Time Division of the National Institute of Standards and Technology in Boulder, Colorado. Procurement of the electronics for the phase-locked loop and for temperature control also occurred during this first year.

The oscillator will be mounted on a cryogenic probe that exists at NASA's Jet Propulsion Laboratory. In 1997, the probe was moved to a laboratory where quieter conditions are expected to prevail and was operated in the new space. Parts were fabricated for the high-resolution thermometer that will be employed in the new temperature-control scheme of the oscillator.

The quality factor of the superconducting microwave cavity is crucial to the development of this ultrastable oscillator. A niobium processing facility has been set up by modifying an ultrahigh vacuum system that exists in a laboratory at the California Institute of Technology. E-beam heating to temperatures near  $2000^{\circ}\text{C}$  in vacuum pressures below  $10^{-9}$  torr will be applied to the niobium to produce the high Qs. New cavities produced by this vacuum processing facility will be tested by the end of calendar year 1997.

### Materials Characterization System

The first year of this task involved adapting an existing cryogenic probe for the measurement of absorption of microwave signals in HTSC materials, and then measuring the absorption in several single-crystal samples produced by collaborators from Boeing. These measurements are continuing so that the best processing procedures for the HTSC materials can be defined. The procedures that evolve will provide the highest Q in the microwave cavity to be applied to the characterization system.

A high-Q cavity will be demonstrated in the second year of this task. A "hot finger" system for placing the sample inside the cavity and sweeping the temperature of the sample while the cavity is kept cold has been designed, and preliminary tests have been performed. The full implementation of this system will be completed in the third year. During the final year of this task, software for analyzing the absorption data will be developed, and certain standard measurements will be recorded.

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## APPLICATION & TECHNOLOGY TRANSFER

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As described above, the ultrastable oscillator will first be utilized to provide a comparison signal for other oscillator systems under development in the microgravity program. Since this oscillator will provide better short-term stability than atomic oscillators can obtain, the superconducting cavity stabilized oscillator can also be tied to an atomic oscillator to obtain a stable frequency source over a wider range of measurement times than either separate system can produce. Together, these oscillator systems will develop into the new state of the art in stable frequency sources.

The materials characterization system with cavity Q greater than a million will be able to detect very low levels of imperfections in materials samples. User-friendly procedures for mounting a sample in the cavity and for measuring the signal absorption over a range of sample temperatures will allow materials developers to quickly check the products of their fabrication techniques. By attaching the cavity system to a small cryocooler and providing fiducial measurements of material standards, this facility should find use in ground-based laboratories as well as on the ISS.

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# Determination of Soot Volume Fraction via Laser-Induced Incandescence

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## OBJECTIVES

Quantitative data obtained by advanced diagnostics are often needed to test detailed predictions in numerical models and to provide new knowledge of microgravity combustion science. In response to this need for advanced diagnostics, and with the recognition of the constraints of a microgravity environment, laser-induced incandescence (LII) is being developed for microgravity combustion research as a two-dimensional imaging diagnostic for the measurement of soot volume fraction. LII, in conjunction with other optical imaging techniques, provides unparalleled temporal and spatial resolution, yielding insights into soot formation and oxidation processes.

Present methods of measuring soot volume fraction are limited to line-of-sight methods. These methods offer poor temporal and spatial resolution and require assumptions about the path length and physical properties of the soot. LII utilizes the spatial and temporal properties of pulsed laser excitation to heat soot to far greater temperatures than ordinary flame temperatures and exploits the resultant blue-shifted incandescent emission from the laser-heated soot. The incandescence is theoretically predicted to be a measure of soot volume fraction, which is the feature of soot that governs many physical processes such as radiative heat transfer from flames. LII offers high temporal resolution by producing signals induced by a single laser pulse. This technique is being extended to two-dimensional imaging applications and has been calibrated against gravimetric sampling so as to be independent of the refractive index of soot.

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## METHOD

Laser-induced incandescence of soot occurs when intense laser light heats the soot to temperatures far above the background. Theoretical analyses in various literature sources describe the interaction of laser radiation with suspended particles. For sub-microsecond laser pulses, an energy balance equation indicates that the energy addition rate greatly exceeds the loss rate from thermal conduction, vaporization, or radiation. For example, the temperature of a soot particle rapidly rises to its vaporization temperature, roughly 4000 K, for laser intensities of  $1 \times 10^7$  W/cm<sup>2</sup> or greater. Thermal calculations show that equilibration of the absorbed energy within the particle occurs rapidly (on the timescale of the laser pulse), but heating of the medium surrounding the particle occurs on a longer timescale. In accord with the Planck radiation law, the

particle thermal emission at these elevated temperatures increases and shifts to the blue compared to the non-laser-heated soot and flame gases.

Measurements have shown that the LII signal is linearly proportional to the soot volume fraction and may be readily interpreted as a relative measure of soot volume fraction. Absolute calibration of the technique may be made by *in-situ* comparison of the LII signal to a system with a known soot volume fraction. Point measurements are easily performed using a photomultiplier tube with spectral and temporal discrimination against natural flame luminosity. One- and two-dimensional imaging measurements may be performed using a gated intensified array camera.

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## SCHEDULE & MILESTONES

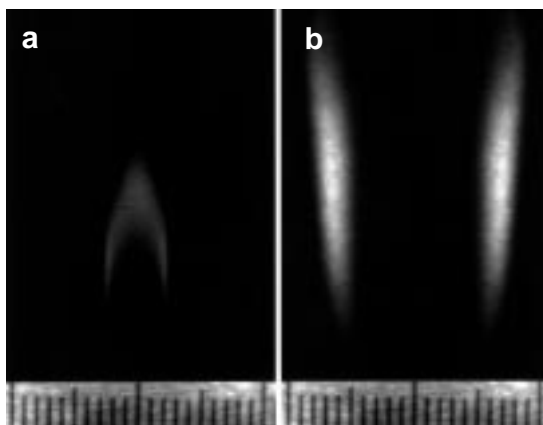
This ATD project was initiated in fiscal year (FY) 1994 and was defined as a four-year effort, to conclude in FY 1997. Accomplishments to date include characterization of the spectral and temporal nature of the LII signal and excitation wavelength dependencies. Linearity between the LII signal and soot volume fraction has also been demonstrated. The technique has been established on a quantitative basis by comparison to light extinction and gravimetric sampling. Current investigations are completing the technique verification milestone by examining photochemical interferences, the effects of soot aggregate composition and size upon the LII signal, and the combination of LII with other comparative diagnostics, such as laser-induced fluorescence. The final milestone of the project, the demonstration of the technique in a reduced-gravity environment, has been completed.

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## APPLICATION & TECHNOLOGY TRANSFER

Soot nucleation and growth processes can be enhanced in a microgravity environment. In normal gravity, buoyant acceleration removes hot combustion products and entrains air, facilitating soot oxidation. In microgravity, hot combustion products remain near the flame front. Radiation from these products and soot contribute to fuel pyrolysis processes, a fundamental step toward soot formation and growth. Without air entrainment or forced convective transport of oxidizer, soot oxidation is limited to convective transport of oxidizer, a far more limiting process than convection. Reduced oxidation rates further increase soot yields.

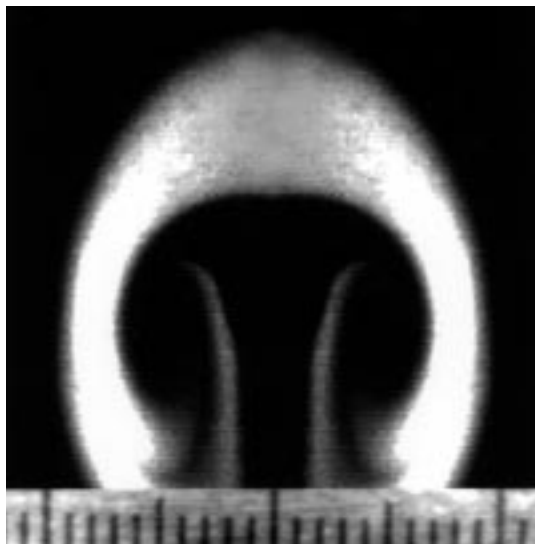
Spatial and temporal scales are also affected by buoyancy-induced convection. In normal gravity, low-density hot gases accelerate and rise from the flame, causing buoyant air entrainment. This in turn causes an inwardly directed radial velocity near the flame sheet, thereby accounting for the radial confinement of the flame. In contrast, in microgravity, the flame spatial extent is determined by a balance between the initial fuel jet flow, its deceleration, and flame stoichiometric requirements. With an increased flame spatial extent in microgravity, the magnitude of spatial gradients in soot concentration is lessened. Increased spatial scales and lowered velocities result in extended temporal scales for resolving soot inception and growth processes.



*Figure 1: LII images of a laminar propane gas-jet diffusion flame (a) in 1 g and (b) in microgravity. The burner nozzle is 11 mm below the picture bottom. The ruler spatial scale is in millimeters.*

An example of these differences between normal-gravity and microgravity environments and the advantages of LII with respect to revealing the extended spatial (and corresponding temporal) scales for studying soot nucleation and growth processes is shown in Figure 1. Figure 1a shows an LII image of a buoyant laminar gas-jet diffusion flame of propane in normal gravity. Figure 1b shows an LII image of the same flame (identical fuel and flow rate) in microgravity. In both environments, the nominal jet Reynolds number was 220, based on the nozzle inner diameter of 1.1 mm and a flow rate of 46 sccm. Each image was obtained with a single laser shot and provides a direct real-time relative soot volume fraction map. Distinct differences are observed in the soot volume fraction spatial distribution and concentration gradients between the normal-gravity and microgravity flames. The extended spatial and temporal scales in the absence of buoyancy-induced convection cause a

higher soot volume fraction in the microgravity flame by roughly a factor of two compared to normal gravity.



*Figure 2: LII image of a vortex formed during the initial formation of a laminar gas-jet flame of propane in microgravity. The burner nozzle is 11 mm below the picture bottom.*

Figure 2 shows an LII image of soot within a vortex formed by propane fuel issuing from a gas-jet nozzle. The central bulge is due to the initial jet momentum, while the side recirculation regions result from air entrainment into the shear layer. Initiation of fuel flow and subsequent ignition were performed in low gravity (after package release from the “music” wire). Identical fuel-flow conditions as for the laminar flame in Figure 1 were used, with the only difference being the amount of time between ignition and image collection. The higher soot volume fraction in the recirculation regions is sensible, as fuel parcels in these regions experience extended times at elevated temperatures, promoting pyrolysis processes and soot growth. Detection and resolution of the transient vortex and its spatial gradients illustrate the temporal and spatial capabilities of LII.

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# Vibration Isolation and Control System for Small Microgravity Payloads

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## OBJECTIVES

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As a research facility for microgravity science, the proposed International Space Station will be used for numerous investigations that will require an active isolation device to provide a quiescent acceleration environment. Such investigations could include experiments in protein crystal growth, combustion, and fluid mechanics. Many of these experiments are especially sensitive to accelerations in the frequency range below 1 Hz. Numerous disturbance sources, including crew activity, fans, pumps, and motors will introduce accelerations significantly larger than the level that can be tolerated by these experiments. The ubiquity of the disturbance sources and the difficulty in characterizing them preclude source isolation; therefore, vibration isolation is required to attenuate the anticipated disturbances to an acceptable level.

## METHOD

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In this ATD effort, the technology developed for microgravity vibration isolation demonstrated by STABLE (Suppression of Transient Accelerations By LEvitation) will be extended for a minimum-volume vibration isolation system for the space station. This technology is intended for use with the Microgravity Science Glovebox (MSG) on the space station, since the MSG currently has no provision for attaining the desired quiescent environment. The glovebox vibration isolation system is called G-LIMIT (GLovebox Integrated Microgravity Isolation Technology). Unique to G-LIMIT is the modularity and small size necessary for rapid turnaround and low cost for multiple users. In addition, this system provides the unique capability of measuring the absolute acceleration, independent of accelerometers, as a by-product of the control system. G-LIMIT will also have the capability of generating pristine accelerations to enhance experiment operations.

The basic objective of a vibration isolation system is to mitigate the accelerations transmitted to an experiment through umbilicals and payload-generated sources. The required attenuation can be derived from the anticipated disturbance environment and required acceleration levels. To provide the desired environment, the isolation system is required to pass through the quasi-steady accelerations while

providing attenuation above 0.01 Hz. At frequencies above 10 Hz, the required attenuation level is -60 dB, or three orders of magnitude of attenuation. Due to the need to provide data, power, vacuum, and fluid resources to the payload, the isolated experiment is physically connected to the base (station rack structure) by an umbilical that tends to be too stiff to provide sufficient passive attenuation. From a structural dynamics perspective, the attenuation requirement may be interpreted as requiring a soft spring connection between the base and the isolated experiment. The isolation system must also reject forces transmitted directly to the experiment (by pumps, fans, motors, etc.). To accomplish this effective softening of the umbilicals while rejecting direct disturbances requires an active isolation system. By sensing relative position, the isolated experiment can follow the very low-frequency motion of the base while attenuating the base motion above 0.01 Hz. High-bandwidth acceleration feedback increases the effective mass of the payload, thereby attenuating the direct disturbance response.

## SCHEDULE & MILESTONES

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The proposed tasks are organized in a three-year plan with the first year generally focused on development of the technology at a component level and the second and third years addressing fabrication and testing of the isolator module and the full isolation system.

During the first year of the ATD project, significant progress has been made with regard to the unique self-sensing position measurement concept, for which a patent application has been submitted. Two new actuator designs that will be optimized for the glovebox isolation system are under way. One concept is being investigated under a grant to the Texas A&M University. A novel concept for electromagnetic actuation, which utilizes a printed circuit card and a pair of magnets for actuation and gap position sensing, is also under development. In addition, a conceptual design has been developed ahead of schedule for the prototype six-degrees-of-freedom isolator for the MSG. Figure 1 illustrates the conceptual design for G-LIMIT. These accomplishments have placed the project ahead of schedule, poised for successful development of a prototype flight vibration isolation system for the MSG.

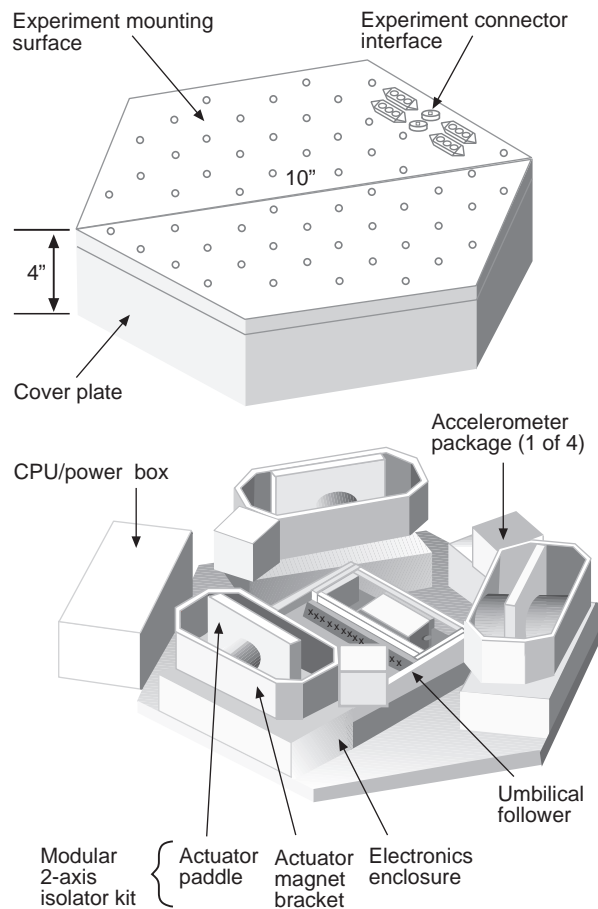
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## APPLICATION & TECHNOLOGY TRANSFER

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The success of STABLE, ARIS (Active Rack Isolation System), and MIM (Microgravity Isolation Mount) has demonstrated the need for microgravity vibration isolation in order to achieve the quiescent environment necessary for conducting microgravity science. However, neither STABLE, ARIS, nor MIM is capable of being used for glovebox experiments, especially experiments performed in the glovebox airlock. The capability for isolating experiments inside the glovebox and airlock will be provided by the new technology developed under this ATD project, uniquely filling a significant niche in the capabilities of the MSG for performing microgravity science.

A significant number of fluids and materials science experiments will benefit from G-LIMIT, and there already exists a need for this type of capability in the protein crystal growth (PCG) area. With the fair number of PCG experiments done on space shuttle flights, the data garnered to date regarding the effect of vibrations on PCG are not conclusive and present a rather confusing picture. A well-characterized (with respect to vibration) PCG experiment is needed to better understand this issue. Similarly, in fluid physics and materials processing experiments, the subtle influences of the acceleration environment are manifest in the results. For example, enhanced diffusion coefficients are routinely used to fit data from semiconductor crystal growth flight experiments. Modeling studies have indicated that these experiments are particularly prone to low-frequency vibrations even of very small amplitude. Pure fluids experiments are also susceptible to low levels of acceleration, especially when buoyancy-causative forces, such as thermal/concentration gradients, are present. The investigations of pure thermal diffusion during the Spacelab–Japan mission and on the second United States Microgravity Laboratory (USML–2) mission also show the contribution of spacecraft accelerations to augmented diffusion. Besides generating buoyant convective forces, these excitations can induce surface jitter, like that noticed in the Surface Tension–Driven Convection Experiment; cause subtle effects in experiments wherein operating parameters are tightly controlled, such as in investigations of critical point phenomena; and cause unexpected particle motion in isothermal fluids, as observed in the Flash Evaporator System experiment on the second International Microgravity



*Figure 1: Schematic of GLovebox Integrated Microgravity Isolation Technology (G-LIMIT)*

Laboratory (IML–2) mission. While all of the results observed in space experiments are not yet understood, the development of vibration isolation will greatly enhance the scientific process by providing a capability that will help gauge the effect of controlling an important parameter in low-gravity experiments.

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# A New Ultrahigh-Resolution Near-Field Microscope for Observation of Protein Crystal Growth

## OBJECTIVES

The primary objective of this ATD project is to build and test a new optical method for observing protein crystals as they nucleate and grow. The new technique will be based on a tapered-fiber probe in a near-field scanning optical microscope (NSOM). Standard microscopy is typically limited to about 1-micron resolution, while the NSOM is capable of resolving 12-nm objects. A state-of-the-art NSOM will be built at Marshall Space Flight Center (MSFC) and then used to visualize individual molecules in a protein crystal growth solution. Labeling the proteins with a fluorescent material and using polarization techniques should allow determination of the molecules' orientation both in the solution and on a crystal surface with the NSOM. New probes will be designed and fabricated to improve the capabilities of the NSOM. The new probe designs may improve the resolution or may eliminate the need for scanning.

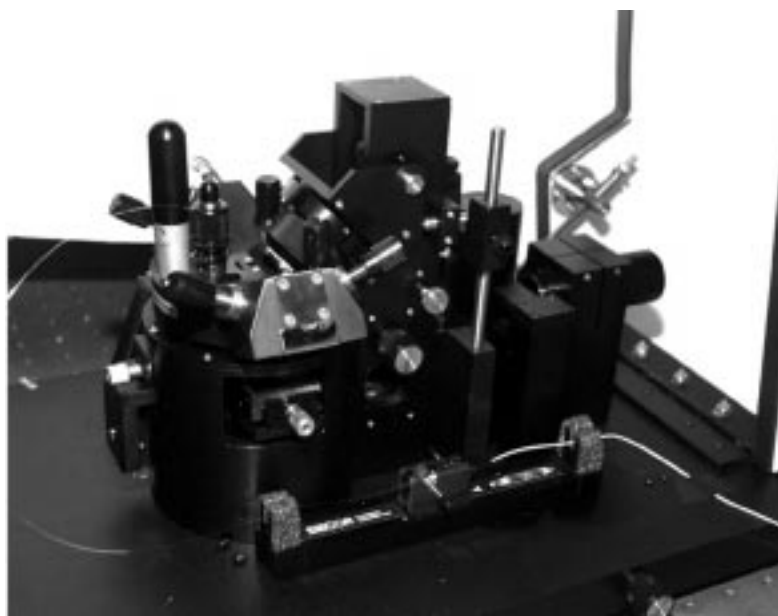


Figure 1: NSOM apparatus

## METHOD

The ATD project will take place at MSFC with assistance provided by the University of Alabama, Huntsville. Both groups have experience in designing and constructing optical systems, and the group at

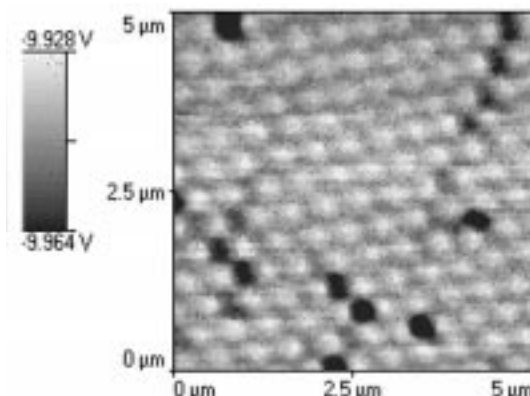


Figure 2: NSOM image of aluminum deposition

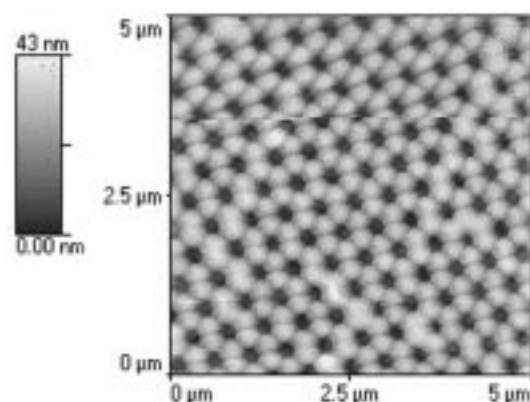


Figure 3: Topography of aluminum deposition

MSFC has extensive experience in protein crystal growth. A near-field scanning optical microscope will be designed and constructed at MSFC, and tapered fibers will be constructed at MSFC with a fiber puller. Coating the fibers with aluminum will take place in a vacuum sputtering system located at MSFC. The tapered-fiber probe will be able to be immersed directly into a protein crystal growth solution. Tunneling near-field optical microscopy will also be examined for possible application to observing protein crystal growth. As each improvement is made in the NSOM system under development, it will be evaluated through use in protein crystal growth experiments. From the results of the experiments, the NSOM system will be refined and improved to further enhance its performance.

Recently, success has been obtained in incorporating the fluorescent probe lucifer yellow into the substrate binding cleft of chicken egg-white

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lysozyme. The covalently modified protein crystallizes with the same habit (tetragonal) as the unmodified protein, and from this it can be concluded that the probe is apparently not interfering with the crystal growth process. The bound lucifer yellow retains its fluorescence. Thus, there is now a suitable fluorescent derivative of the most commonly used model material in protein crystal growth studies. Ongoing work in this laboratory in conjunction with other projects will concentrate on incorporation of other fluorescent probes at the same site to further expand the range of experimental possibilities. With suitably labeled molecules and a functional NSOM, the following types of experiments can be done: flow effects, aggregate sizing, cluster formation on crystal surfaces, and poisoned surfaces.

Attempts at improving the quality of the tapered-fiber probe will involve experimenting with fiber pulling and coating techniques. In addition, investigations will be conducted in making novel fiber probes.

Fluorescent labels will be applied to the proteins and experiments conducted using each development level of the NSOM. Experiments involving polarization detection to determine the orientation of protein molecules on protein crystal surfaces will be possible with even a low-resolution NSOM. It is anticipated that several flight experiments will develop from this ATD project.

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## **SCHEDULE & MILESTONES**

A commercial NSOM has been constructed at MSFC, and a fiber puller has been purchased to construct tapered-fiber probes. The first probes will be standard tapered-fiber probes built according to current techniques. Measuring the capabilities (i.e., resolution, scanning area, etc.) of the NSOM has begun. Once the capabilities are known, protein crystals will be measured with the system.

At least 11 different fluorescent derivatives of lysozyme have been synthesized to date. There are two reasons for making many derivatives. First, different probes have different changes in their fluorescent behavior between free-in-solution and covalently bound states. Those that have the highest fluorescence signal in the bound state are the most desirable. Second, the probes are generally not rigidly bound; they have a motion that is independent of the proteins. When doing polarization studies, it is

important that the probe absolutely determines the underlying protein's orientation. This can only be done if the probe is rigidly bound. A derivative has recently been made that, on the basis of the initial characterization results, appears to be rigidly bound.

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## **APPLICATION & TECHNOLOGY TRANSFER**

The closest analogy to near-field scanning optical microscopy is atomic force microscopy, which has rapidly become a valuable research tool in many areas of science and technology. While NSOM technology will be developed for use with protein crystal growth in this project, it is anticipated that many other areas of science will have an immediate use for this technology. The utility of NSOM technology should also extend past that of imaging surfaces to allow the "imaging" of small volumes of solutions. In a suitably designed system, this will allow the direct counting and sizing of aggregate species present, eliminating the problems in resolving such information from polydisperse solutions by methods such as light scattering. Both topics, surface studies and particle sizing of solutions, represent areas of high commercial potential with the development of suitably improved instrumentation or methods.

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## Appendix C

### Abbreviations and Acronyms

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AADSF	Advanced Automated Directional Solidification Furnace
AC	alternating current
ACMC	alternating current modulation calorimetry
AOTF	acousto-optic tunable filter
ARIS	Active Rack Isolation System
ATD	Advanced Technology Development
BRS	Bioproduct Recovery System
BSO	bismuth silicon oxide
BTF	Biotechnology Facility
CAB	copper ammonium bromide
CGF	Crystal Growth Furnace
CHeX	Confined Helium Experiment
CRADA	Cooperative Research and Development Agreement
CW	continuous wave
DC	direct current
DSP	Digital Signal Processing
DYNAMX	Critical Dynamics in Microgravity Experiment
EMI	electromagnetic interference
ESA	European Space Agency
ESL	electrostatic levitation
FM	frequency-modulated
FTIR	Fourier transform infrared
FVS	Free-Vortex Separator
FY	fiscal year
G-LIMIT	GLovebox Integrated Microgravity Isolation Technology
HEDS	Human Exploration and Development of Space
HRT	high-resolution thermometer
HTESL	High-Temperature Electrostatic Levitator
HTSC	high-temperature superconductor
IML	International Microgravity Laboratory
ISS	International Space Station
JPL	Jet Propulsion Laboratory
JSC	Johnson Space Center
LeRC	Lewis Research Center
LFI	laser-feedback interferometry
LII	laser-induced incandescence
LMD	Liquid Metal Diffusion
LMS	Life and Microgravity Spacelab
LPE	Lambda Point Experiment
LTMPF	Low-Temperature Microgravity Physics Facility
MEMC	Monsanto Electronic Materials Corporation
MGHPF	Moving-Gradient Heat Pipe Furnace
MGM	Mechanics of Granular Materials
MIM	Microgravity Isolation Mount



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MRD	Microgravity Research Division
MRP	Microgravity Research Program
MSFC	Marshall Space Flight Center
MSG	Microgravity Science Glovebox
NEI	National Eye Institute
NIH	National Institutes of Health
NRA	NASA Research Announcement
NSOM	near-field scanning optical microscope
NTR	New Technology Report
OLMSA	Office of Life and Microgravity Sciences and Applications
PCG	protein crystal growth
PCS	Passive Culture System
PDT	penetration depth thermometer
PI	principal investigator
PIV	particle image velocimetry
RF	radio frequency
SBIR	Small Business Innovative Research
SDLE	Self-Diffusion in Liquid Elements
SET	single-electron transistor
SLS	surface light scattering
SQUID	Superconducting Quantum Interference Device
STABLE	Suppression of Transient Accelerations by LEvitation
STEP	Satellite Test of the Equivalence Principle
STS	Space Transportation System
TCA	Technology Cooperation Agreement
TII	Theta Industries, Inc.
TSA	two-stage SQUID amplifier
TSSA	two-stage series array SQUID amplifier
UF	Utilization Flight
USML	United States Microgravity Laboratory
USMP	United States Microgravity Payload
VPS	vacuum plasma spray
XTM	X-Ray Transmission Microscope



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